

Studies on organolanthanide complexes.

LVIII [☆]. Syntheses of 1,1'-(3-oxa-pentamethylene) dicyclopentadienyl divalent organolanthanides (Ln = Sm, Yb) and X-ray molecular structure of O(CH₂CH₂C₅H₄)₂Yb(DME)Changtao Qian ^{a,*}, Chengjian Zhu ^a, Yonghua Lin ^b, Yan Xing ^b^a Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Lu, Shanghai 200032, China^b Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, 109 Stalin Street, Changchun 130022, China

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Abstract

The compounds O(CH₂CH₂C₅H₄)₂Ln(THF)₂ [Ln = Sm(1), Yb(2)] were synthesized by the reduction of O(CH₂CH₂C₅H₄)₂LnCl with sodium metal in tetrahydrofuran (THF) at room temperature. Recrystallization of 2 from dimethoxyethane (DME) produced the single-crystal O(CH₂CH₂C₅H₄)₂Yb(DME) (3) whose structure has been determined by an X-ray diffraction study. The crystals are orthorhombic, space group *Pcab*, with *a* = 14.168(4), *b* = 13.541(6), *c* = 19.314(8) Å, *Z* = 8, *D*_{calc.} = 1.66 g cm⁻³.

Keywords: Samarium; Ytterbium; Cyclopentadienyl; Crystal structure; IR spectroscopy; Mass spectrometry

1. Introduction

In recent years, study of the complexes of metals that are in the +2 oxidation state has yielded particularly remarkable and striking results in the case of the organometallic chemistry of organolanthanoids [1–9]. The major breakthrough in the chemistry of divalent organolanthanides in general, and that of Sm^{II} in particular, involved the use of pentamethylcyclopentadienyl which confers stability, solubility and crystallinity on organometallic compounds. Unlike cyclopentadienyl or methylcyclopentadienyl compounds, the pentamethylcyclopentadienyl derivatives of Sm^{II} are soluble in aromatic and coordinating solvents. Moreover, (C₅-Me₅)₂Sm-(THF)₂ is capable of unusual transformations of multiple-bonded substrates [6,10].

The useful properties bestowed by pentamethylcyclopentadienyl may be attributed to its large size [11,12]. Any such group may be also expected to offer similar

properties. It was demonstrated [3] that the derivatives of Sm^{II} could also be prepared using bulky *t*-butyl-substituted cyclopentadienyl as ligands. The effect of C₅Me₅ ligands and ^tBuC₅H₄ is to decrease the acidity of divalent organolanthanide complexes. This effect might also be achieved by using more accessible cyclopentadienyl ligands with donor substituents. Moreover, the early research results of our group have demonstrated that the effect of the methoxyethylcyclopentadienyl ligand is similar to that of C₅Me₅ and ^tBuC₅H₄ in the synthesis of low-valent organolanthanide complexes, and that ring-bridged dicyclopentadienyl ligands such as [C₅H₄(CH₂)_{*n*}C₅H₄]²⁻ (*n* = 3, 5), [C₅H₄(CH₂)₂O(CH₂)₂C₅H₄]²⁻, [C₅H₄(CH₂)₂N-(CH₃)(CH₂)₂C₅H₄]²⁻, etc. can be used to prepare stable dicyclopentadienyl early lanthanide halides and decrease the torsional mobility of the cyclopentadienyl rings [13,14].

Herein, we would like to report the syntheses of oxygen-containing bridged-dicyclopentadienyl divalent lanthanide complexes O(CH₂CH₂C₅H₄)₂Yb(THF)₂ (Ln = Sm, Yb) and the X-ray molecular structure of O(CH₂CH₂C₅H₄)₂Yb(DME).

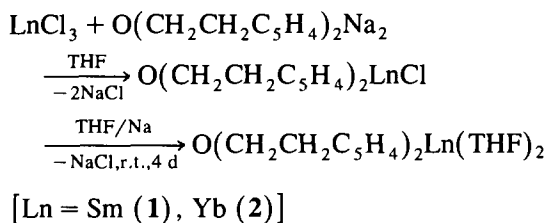
[☆] For Part LVII, see Ref. [23].

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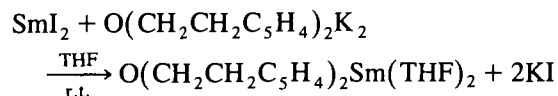
2. Results and discussion

2.1. Syntheses

The interaction of LnCl_3 ($\text{Ln} = \text{Sm}, \text{Yb}$) and 1,1'-(3-oxa-pentamethylene)cyclopentadienyl disodium salt in THF provided trivalent complexes of this type $\text{O}(\text{CH}_2\text{CH}_2\text{C}_5\text{H}_4)_2\text{LnCl}$ ($\text{Ln} = \text{Sm}, \text{Yb}$) [15]. The chlorides were then reduced with sodium metal in THF at room temperature for 3 d, purple–black ($\text{Ln} = \text{Sm}$) and red–black ($\text{Ln} = \text{Yb}$) solutions were being formed, respectively. Following precipitation by the addition of n-hexane and drying in vacuum, new divalent organolanthanide complexes, i.e. the purple compound $\text{O}(\text{CH}_2\text{CH}_2\text{C}_5\text{H}_4)_2\text{Sm}(\text{THF})_2$ and the red–orange compound $\text{O}(\text{CH}_2\text{CH}_2\text{C}_5\text{H}_4)_2\text{Yb}(\text{THF})_2$, were obtained in good yield:



Another method was also used to synthesize compound 1. Thus, SmI_2 reacted with $\text{O}(\text{CH}_2\text{CH}_2\text{C}_5\text{H}_4)_2\text{K}_2$ in THF at room temperature to give compound 1 in good yield:



Compounds 1 and 2 are soluble in THF and DME, but insoluble in aromatic and aliphatic solvents such as benzene, toluene and hexane. Compound 2 dissolves in DME to give a red–green solution. Recrystallization from DME gave red single crystals of $\text{O}(\text{CH}_2\text{CH}_2\text{C}_5\text{H}_4)_2\text{Yb}(\text{DME})$ (3).

2.2. Spectral analysis

The EI mass spectra of the two complexes revealed the THF-free parent molecular ion $[\text{M} - 2\text{THF}]^+$ and related fragments as well as the fragments of coordinated THF. No fragment greater than $[\text{M}]^+$ was detected, thus suggesting that the complexes are monomeric and that the coordinated THF is released at first.

The IR spectra of complexes 1 and 2 were measured from 4000 to 200 cm^{-1} . Characteristic absorption peaks due to cyclopentadienyl are evidently present [13], with the C–H stretching vibration of Cp in complex 2 (3054 cm^{-1}) being shifted to lower frequency as compared with the corresponding trivalent complex $\text{O}(\text{CH}_2\text{CH}_2\text{C}_5\text{H}_4)_2\text{YbCl}$ (3080 cm^{-1}) [15].

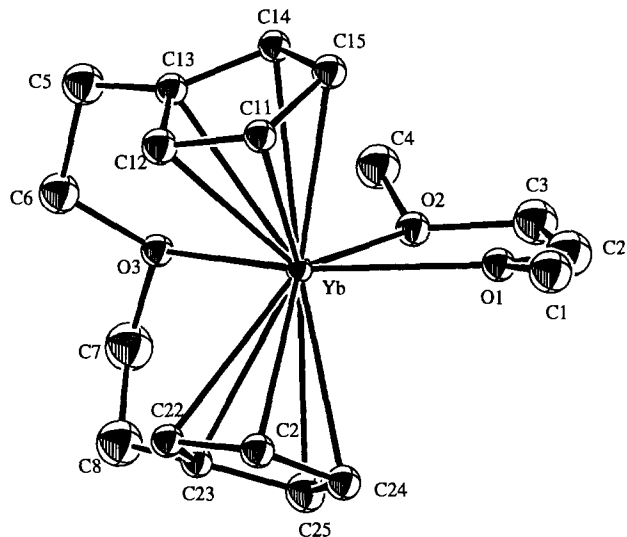


Fig. 1. ORTEP drawing of the molecular structure of $\text{O}(\text{CH}_2\text{CH}_2\text{C}_5\text{H}_4)_2\text{Yb}(\text{DME})$ with the numbering scheme.

The ^1H NMR spectrum of the diamagnetic Yb^{II} complex 2 exhibited two sharp singlets at δ 5.65 and 5.43 ppm, and the $\Delta\delta$ value of 0.22 ppm implying that the torsional mobility of the cyclopentadienyl rings is lower [16]. The OCH_2 signals (δ 3.67 ppm) in the bridge chain shifted to lower fields compared with those of the disodium salt, these results showing that an intramolecular coordination bond ($\text{Ln} \leftarrow \text{O}$) is formed. The chemical shifts of the α - and β -protons of the coordinated THF molecules were found at δ 3.70 and 1.84 ppm, respectively.

2.3. Molecular structure of $\text{O}(\text{CH}_2\text{CH}_2\text{C}_5\text{H}_4)_2\text{Yb}(\text{DME})$

The crystal structure of compound 3 consists of discrete molecules without short contacts. Fig. 1 depicts the structure of the complex.

The formal coordination number of the central metal is nine and the coordination geometry around the ytterbium ion can be described as an approximate trigonal bipyramid, in which the apices are O(3) and O(1), and the apices of the triangle are cent(1), cent(2) and O(1), where cent(1) and cent(2) are the centroids of the cyclopentadienyl rings C(11)–C(15) and C(21)–C(25), respectively. The coordinate oxygen atoms O(1), O(2), O(3) and the ytterbium ion are in the same plane. Selected bond lengths and angles are listed in Table 3 below.

The angle cent(1)–Yb–cent(2) has a value of 132.7° which is within the expected range and agrees well with those in analogous dicyclopentadienylytterbium derivatives such as $(\text{C}_5\text{H}_5)_2\text{Yb}(\text{DME})_2$ (133.9°) [17], $(\text{Me}_3\text{SiC}_5\text{H}_4)_2\text{Yb}(\text{THF})_2$ (133°) [18], $(\text{CH}_2)_3(\text{C}_5\text{H}_4)_2\text{Yb}(\text{THF})_2$ (127°) [4], $(\text{CH}_3\text{OCH}_2\text{CH}_2\text{C}_5\text{H}_4)_2\text{Yb}(\text{THF})_2$ (128.4°) [9], $(^t\text{BuC}_5\text{H}_4)_2\text{Yb}(\text{THF})_2$ (134°) [5] and $(\text{C}_5\text{Me}_5)_2\text{Yb}(\text{NH}_3)(\text{THF})$ (143.5°) [19], but obviously

smaller than in the solvent-free $(C_5Me_5)_2Yb$ (158°) [20]. The angle $O(1)=Yb=O(2)$, which has a value of 66.4° , is smaller than that in $(C_5H_5)_2Yb(DME)$ (69.3°) [16]. This is due to the oxygen in the bridge chain coordinating to the central metal and thus increasing the steric crowding around the Yb ion.

The ytterbium–carbon distance ranges from 2.665(15) to 2.765(17) Å, values which are comparable with those in the dicyclopentadienylytterbium(II) derivatives. The $Yb=O(DME)$ distances of 2.468 and 2.494 Å are similar to those of complexes such as $(^tBuC_5H_4)_2Yb(THF)_2$ (2.430 Å) [5], $(Me_3SiC_5H_4)_2Yb(THF)_2$ (2.410 Å) [18], $(C_5Me_5)_2Yb(THF)$ (2.41 Å) [21], $(CH_2)_3(C_5H_4)_2Yb(THF)_2$ (2.42 Å) [4], $(C_5Me_5)_2Yb(NH_3)(THF)$ (2.46 Å) [19], $(C_5H_5)_2Yb(DME)$ (2.466 Å) [17] and $(CH_3OCH_2CH_2C_5H_4)_2Yb(THF)$ (2.496 Å) [9]. The bond length of the bridged oxygen atom associated with the ytterbium ion is 2.488 Å, and is in the range mentioned above.

3. Experimental details

The compounds described are extremely air- and moisture-sensitive, so the syntheses and subsequent manipulations were performed by use of Schlenk techniques under dried oxygen-free argon. All solvents were refluxed and distilled either over finely divided $LiAlH_4$ or over blue sodium benzophenone under argon immediately before use. Anhydrous lanthanide chlorides were prepared from the pure oxides. SmI_2 was prepared from Sm metal and I_2 [22]. $O(CH_2CH_2C_5H_4)_2M_2$ ($M = Na, K$) were prepared by the reaction of $O(CH_2CH_2C_5H_4)_2$ with the metals [15].

Melting point and thermal decomposition temperatures were determined in sealed argon-filled capillaries and were uncorrected. Mass spectra were recorded on a Finnigan 4021 spectrometer. 1H NMR spectra were obtained on a Varian XL-200 spectrometer referenced to external Me_4Si . $THF-d_8$ was dried over Na/K alloy and degassed by freeze–thaw cycles on a vacuum line. Infrared spectra were recorded on a Perkin-Elmer 983 IR spectrometer with Nujol and Fluorolurbe mulls and were examined between disk-shaped CsI crystals or polyethylene plates. Analyses of rare earth metals in complexes were accomplished using a direct complexometric titration procedure with disodium EDTA. Carbon and hydrogen analyses were performed by combustion method in an aluminium tube.

3.1. Preparation of $O(CH_2CH_2C_5H_4)_2Sm(THF)_2$ (1)

(a) From $SmCl_3$

A tetrahydrofuran solution containing 1 equiv. of 1,1'-(3-oxa-pentamethylene)dicyclopentadienyl disodium salt was added slowly to 1.24 g (4.83 mmol) of

anhydrous $SmCl_3$ in 50 ml of THF at $-25^\circ C$, then allowed to warm up to ambient temperature and stirred overnight to give a yellow solution. The Schlenk flask was centrifuged, the precipitate separated and the solution reduced in volume to about 20 ml. Excess sodium sand was added to the solution and the mixture stirred for 4 d at room temperature, whereupon the colour of the solution gradually turned to purple–black. The THF solution was separated from the precipitate and reduced to about 10 ml. Addition of 20 ml of n-hexane gave a purple–black solid, which was then dried in vacuum at room temperature. The weight of the solid was 1.60 g, yield 67%; m.p. $162^\circ C$ (dec.). Anal. Found: C, 52.60; H, 5.68; Sm, 30.97%. $C_{22}H_{32}O_2Sm$ Calc.: C, 53.39; H, 6.52; Sm, 30.40%. IR (cm^{-1}): 3062 (s); 2926 (s); 2862 (m); 2726 (w); 1608 (w); 1460 (w); 1454 (w); 1432 (m); 1375 (m); 1272 (w); 1246 (w); 1193 (m); 1100 (m); 1071 (s); 1038 (s); 1010 (m); 973 (m); 791 (m); 755 (s); 673 (m); 624 (m); 600 (m); 541 (m); 486 (w); 342 (s). MS m/z : 352 $[M - 2THF]^+$; 202 $[M - Sm]^+$; and 72 $[THF]^+$.

(b) From SmI_2

Complex **1** was also produced by the reaction of SmI_2 and $O(CH_2CH_2C_5H_4)_2K_2$ in equivalent amounts in THF. $O(CH_2CH_2C_5H_4)_2K_2$ (3 mmol) in 10 ml of THF was added to SmI_2 (3 mmol) in 30 ml of THF at room temperature. The solution turned purple and was stirred overnight. The Schlenk flask was centrifuged to give the THF solution, which was reduced in volume to about 10 ml. Addition of 20 ml of n-hexane gave a product which was dried in vacuum at room temperature to afford a purple–black solid (**1**): 0.91 g, yield 61.6%. Anal. Found: C, 53.19; H, 5.72; Sm, 30.70%. $C_{22}H_{32}O_2Sm$ Calc.: C, 53.39; H, 6.52; Sm, 30.40%.

3.2. Preparation of $O(CH_2CH_2C_5H_4)_2Yb(THF)_2$ (2)

Method (a) for complex **1**, described above, was used with 1.33 g (4.76 mmol) of $YbCl_3$ and gave 1.81 g (yield 73.5%) of a red–yellow solid of complex **2**; m.p. $144^\circ C$ (dec.). Anal. Found: C, 51.06; H, 5.74; Yb, 33.33%. $C_{22}H_{32}O_2Yb$ Calc.: C, 51.06; H, 6.23; Yb, 33.44%. IR (cm^{-1}): 3054 (s); 2934 (s); 2861 (s); 2729 (m); 1731 (w); 1601 (m); 1521 (m); 1478 (w); 1463 (w); 1459 (s); 1430 (m); 1424 (m); 1372 (s); 1272 (m); 1187 (m); 1164 (m); 1111 (s); 1070 (s); 1039 (s); 1023 (s); 972 (s); 947 (s); 928 (s); 896 (s); 809 (m); 742 (s); 723 (m); 675 (s); 591 (m); 374 (s). 1H NMR ($THF-d_8$) δ : 5.65 (s, 4H); 5.43 (s, 4H); 3.70 (m, 8H); 3.67 (m, 8H); 2.75 (m, 4H); 1.84 (m, 8H) ppm. MS m/z : 374 $[M - 2THF]^+$; 202 $[M - Yb]^+$; 72 $[THF]^+$.

3.3. Preparation of $O(CH_2CH_2C_5H_4)_2Yb(DME)$ (3)

The red–yellow crystals of complex **3** were obtained by recrystallizing **2** in dimethoxyethane (DME) at room

Table 1
Crystallographic data for $O(CH_2CH_2C_5H_4)_2Yb(DME)$

Compound	$YbC_{18}H_{26}O_3$
Molecular weight	463.43
Crystal system	orthorhombic
Space group	$Pcab$
Cell constants	
a (Å)	14.186(4)
b (Å)	13.541(6)
c (Å)	19.314(8)
Cell volume (Å ³)	3710(2)
Density (g cm ⁻³)	1.66
Absorption coefficient (cm ⁻¹)	53.1
Molecular/unit cell	8
$F(000)$	1824
Radiation	Mo K α
Crystal dimension (mm)	$0.24 \times 0.52 \times 0.36$
2θ range (°)	3–52
h, k, l	0–18, 0–19, 0–24
Observed reflections	1924
R	0.049
R_w	0.048
Temperature (°C)	20

temperature. Anal. Found: Yb, 37.80%. $C_{18}H_{26}O_3Yb$
Calc.: Yb, 37.34%.

3.4. X-Ray crystal structure determination

A red–yellow single crystal with approximate dimensions $0.24 \times 0.52 \times 0.36$ mm was sealed in a thin-walled glass capillary under argon. Intensity data were collected at 20°C with a Nicolet R3M/E four-circle

diffractometer using graphite-monochromated Mo K α radiation. Details of the data collection and refinement are listed in Table 1. A total of 2394 unique reflections were measured within the range $0^\circ < 2\theta < 52^\circ$ with $0 \leq h \leq 18$, $0 \leq k \leq 19$, $0 \leq l \leq 24$. Corrections for Lorentz polarization and absorption were applied to the reflection data. The structure was solved by the Patterson method and by difference Fourier synthesis. All positional parameters and temperature factors for non-H

Table 2
Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) with estimated standard deviation (e.s.d.s) in parentheses

Atom	x	y	z	U_{eq}^a
Yb	2100(1)	6263(1)	6344(1)	56(1)
O(1)	756(6)	6427(7)	7148(4)	89(4)
O(2)	781(1)	5099(7)	6093(5)	94(4)
C(1)	691(12)	6997(15)	7723(8)	145(9)
C(2)	-29(17)	5899(17)	7021(12)	174(13)
C(3)	-10(13)	5208(22)	6632(10)	160(13)
C(4)	874(14)	4168(11)	5771(11)	156(10)
C(11)	3311(9)	6222(12)	7415(7)	84(5)
C(12)	3852(9)	6016(12)	6832(7)	103(7)
C(13)	3566(10)	5098(12)	6622(7)	82(6)
C(14)	2885(10)	4757(12)	7055(7)	81(6)
C(15)	2743(9)	5446(12)	7569(7)	95(7)
O(3)	2706(7)	5361(7)	5315(4)	84(4)
C(5)	3925(12)	4588(12)	5964(9)	137(9)
C(6)	3661(14)	5093(14)	5313(8)	128(9)
C(7)	2477(20)	5737(15)	4655(8)	184(16)
C(8)	2518(21)	6743(14)	4586(7)	170(13)
C(21)	2405(13)	8228(13)	6197(8)	94(7)
C(22)	2852(11)	7777(9)	5628(6)	85(6)
C(23)	2231(13)	7386(10)	5195(7)	81(6)
C(24)	1473(13)	8134(12)	6087(10)	96(7)
C(25)	1350(15)	7617(13)	5444(10)	126(9)

^a Equivalent isotropic U defined as one-third of the trace of the orthogonalized V_{ij} tensor.

Table 3
Selected bond lengths (Å) and bond angles (°) with e.s.d.s in parentheses^a

Yb–O(1)	2.468	Yb–O(2)	2.494
Yb–C(11)	2.689	Yb–C(12)	2.679
Yb–C(13)	2.665	Yb–C(14)	2.699
Yb–C(15)	2.765	Yb–O(3)	2.488
Yb–C(21)	2.710	Yb–C(22)	2.694
Yb–C(23)	2.696	Yb–C(24)	2.730
Yb–C(25)	2.742	Yb–cen(1)	2.435
Yb–cen(2)	2.448		
O(1)–Yb–O(2)	66.4	O(1)–Yb–O(3)	144.4
O(2)–Yb–O(3)	78.0	cen(1)–Yb–cen(2)	132.7
O(3)–Yb–cen(1)	92.1	O(3)–Yb–cen(2)	91.9
O(1)–Yb–cen(1)	100.9	O(1)–Yb–cen(2)	102.7
O(2)–Yb–cen(1)	111.3	O(2)–Yb–cen(2)	115.7
C(11)–Yb–C(21)	90.0	C(12)–Yb–C(22)	84.7
C(13)–Yb–C(23)	116.5	C(14)–Yb–C(24)	158.2
C(15)–Yb–C(25)	159.7	O(3)–Yb–C(13)	66.5
O(3)–Yb–C(23)	66.1	C(5)–C(13)–Yb	111.0
C(6)–C(5)–C(13)	114.3	O(3)–C(6)–C(5)	111.3
C(6)–O(3)–Yb	117.5	Yb–cen(1)–C(11)	89.9
Yb–cen(1)–C(12)	88.3	Yb–cen(1)–C(913)	88.4
Yb–cen(1)–C(14)	90.1	Yb–cen(1)–C(15)	93.2
C(11)–C(12)–C(13)	104.9	C(12)–C(13)–C(14)	109.8
C(13)–C(14)–C(15)	108.4	C(11)–C(15)–C(14)	106.3
C(12)–C(11)–C(15)	110.5		

^a The abbreviations cen(1) and cen(2) correspond to the centroids of the cyclopentadienyl rings C(11)–C(15) and C(21)–C(25), respectively.

atoms were refined anisotropically by the full-matrix least-squares technique. Final R and R_w values were 0.049 and 0.048, respectively, for 1924 observed reflections. All calculations were carried out by means of an Eclipse S/140 computer with the SHELXTL computer program system. Scattering factors were taken from the *International Tables for X-ray Crystallography* (1974). Table 2 lists the final atomic positional and thermal parameters, while Table 3 gives the selected bond lengths and bond angles for complex **3** together with the estimated standard deviations.

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