Synthesis and X-ray crystal structure of bis[η5-1,3-bis(trimethylsilyl)cyclopentadienyl] lanthanide and yttrium iodide complexes

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Abstract

Treatment of $[\{(Me_3Si)_2C_5H_3\}_2LnCl]_2$ with excess amounts of NaI in dry THF, or reaction of a mixture of $LnCl_3$ and NaI with two equivalents of $[(Me_3Si)_2C_5H_3]Na$ in dry THF at room temperature, gave five new monomeric complexes $[(Me_3Si)_2C_5H_3]_2LnI(THF)$ (Ln = La (1), Sm (2), Y (3), Er (4), Lu (5)) in 50–69% yield. The mixed π-ligand complex $[\{(Me_3Si)_2C_5H_3\}\{(Me_3Si)C_5H_4\}]LuI(THF)$ (6) was also prepared in a similar manner. The compounds were fully characterized by elemental analyses, IR and MS spectroscopy, ^1H NMR (for diamagnetic metals) and single-crystal X-ray diffraction studies (except 1). In each compound, the C_5 ring centroids, the THF oxygen atom and iodide ligand describe a distorted tetrahedral geometry around the metal. The variations in the metal–iodide bond distances are not as predictable as those in metal–carbon and metal–oxygen bond distances. © 1997 Elsevier Science S.A.

Keywords: Organolanthanide; Crystal structure; Halide exchange; Cyclopentadienyl

1. Introduction

Organolanthanide iodide complexes have their unique chemical properties due to the more labile property of the Ln–I bond. For instance, the reaction of organolanthanide iodide with silver(I) salt of tetraphenylborate gave the organolanthanide cation in high yield [1]; treatment of organolanthanide iodide with $AgSbF_6$ produced the corresponding fluoride complex [2]. Similar reactions with the closely related organolanthanide chlorides are, however, very complicated [3,4]. In this respect, an organolanthanide iodide complex is a better precursor than its chloride analogue. On the other hand, in contrast with the chemistry of organolanthanide chloride complexes, the iodo derivatives are much less studied, presumably due to lower availability and the expensiveness of the starting materials LnI_3 (for recent reviews on organolanthanide chemistry, see Ref. [5]). Several synthetic methods of organolanthanide iodo complexes, including the metathesis of LnI_3 with anionic ligands [1,6–9], reactions of organolanthanide hy-drides or low-valent organolanthanide complexes with organic iodo compounds [10–13] and halide exchange reaction of organolanthanide chlorides with NaI [14], have been reported in the literature. Among these, the last is the most convenient but the least studied. Furthermore, in Cp'' (Cp'' = $1,3-(Me_3Si)_2C_5H_3$) systems, only La and Ce iodo complexes appeared in the literature, without structural data to our knowledge [1], in spite of the fact that the chloro analogues were known for the whole lanthanide series [15–17]. We report herein the systematic investigations of the halide exchange reactions between $[Cp''_2LnCl]_2$ or $[(Cp'')Cp'LnCl]_2$ and NaI in THF, which led to the isolation of X-ray quality crystals of the six new iodide complexes of the type $Cp''_2LnI(THF)$ [Ln = La (1), Sm (2), Y (3), Er (4), Lu (5)] and $(Cp'')Cp'LuI(THF)$ (6) [Cp' = $(Me_3Si)C_5H_4$]. X-ray crystal structures of 2–6 have been determined. Since these are the first organolanthanide iodide complexes with Cp'' ligands to be structurally characterized, they provide useful, basic structural data in the rapidly growing area of organolanthanide chemistry [5]. Taken together, these results allow detailed structural comparisons to be made on the basis of both the metal and ligands present. The syn-
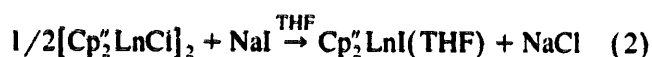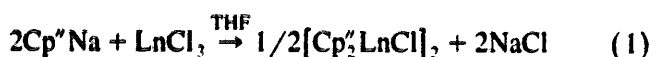
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thetic routes used to obtain these complexes are also informative with respect to organolanthanide halide chemistry.

2. Results and discussion

2.1. Synthesis

Treatment of anhydrous $LnCl_3$ with two equivalents of $Cp''Na$ in THF gave a complex of the type $[Cp''_2LnCl]_2$ [15–17], followed by reaction with excess NaI at room temperature to produce the organolanthanide iodide complex $Cp''_2LnI(THF)$ in 50–69% yield (Eqs. (1) and (2)).

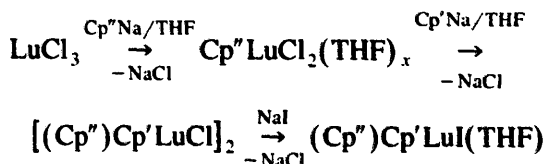$$2Cp''Na + LnCl_3 \xrightarrow{THF} 1/2[Cp''_2LnCl]_2 + 2NaCl \quad (1)$$

$$1/2[Cp''_2LnCl]_2 + NaI \xrightarrow{THF} Cp''_2LnI(THF) + NaCl \quad (2)$$

$Ln = La (1), Sm (2), Y (3), Er (4), Lu (5)$

These iodide complexes can also be prepared in an alternate way by adding $Cp''Na$ to the mixture of $LnCl_3$ and NaI in THF at room temperature (Eq. (3)).

$$2Cp''Na + LnCl_3 + NaI \xrightarrow{THF} Cp''_2LnI(THF) + 3NaCl \quad (3)$$
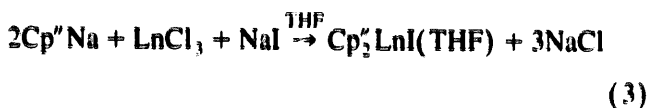
This one-pot synthetic method is more convenient and

less time consuming. The mixed π-ligand lutetium iodide, $(Cp'')Cp'LuI(THF)$ (6), was also prepared in an analogous manner as shown below, but the overall yield is substantially lower than those containing two Cp'' ligands, presumably due to the multi-step synthesis.

$$LuCl_3 \xrightarrow[-NaCl]{Cp''Na/THF} Cp''LuCl_2(THF)_x \xrightarrow[-NaCl]{Cp'Na/THF}$$

$$[(Cp'')Cp'LuCl]_2 \xrightarrow[-NaCl]{NaI} (Cp'')Cp'LuI(THF)$$

The more polarizable iodide ion and more soluble NaI than NaCl in THF may be the driving forces of this reaction. These results show that the halide exchange reaction should be a general method to prepare organolanthanide iodide complexes.

On heating $Cp''_2LnI(THF)$ at 50 °C under vacuum for some hours, the complex with empirical formula Cp''_2LnI was obtained. It is suggested to have a dimeric structure similar to its chloro analogues [15,17]. This unsolvated dimer can readily dissolve in THF to give one THF molecule coordinated monomeric complex of the type $Cp''_2LnI(THF)$.

2.2. Structure

As is evident from the unit cell parameters (Table 1), complexes 2–5 are isomorphous and isostructural. Each crystallizes in space group $P2_12_12_1$ with $Z = 4$. After

Table 1
Crystal data and details of data collection and structure refinement for $Cp''_2LnI(THF)$ and $(Cp'')Cp'LuI(THF)$

Compound	Sm (2)	Y (3)	Er (4)	Lu (5)	(Cp'')Cp'LuI(THF) (6)
Empirical formula	$C_{26}H_{50}IOSi_4Sm$	$C_{26}H_{50}IOSi_4Y$	$C_{26}H_{50}IOSi_4Er$	$C_{26}H_{50}IOSi_4Lu$	$C_{21}H_{42}IOSi_3Lu$
Crystal size (mm³)	0.3 × 0.4 × 0.5	0.2 × 0.25 × 0.8	0.2 × 0.24 × 0.6	0.2x0.2 × 0.7	0.5 × 0.5 × 0.6
M	768.3	706.8	785.2	792.9	720.7
Crystal class	orthorhombic	orthorhombic	orthorhombic	orthorhombic	monoclinic
Space group	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_1/c$
a (Å)	11.023(2)	10.992(2)	10.964(1)	10.944(2)	11.388(1)
b (Å)	16.278(3)	16.268(3)	16.191(1)	16.140(3)	14.202(1)
c (Å)	20.086(4)	20.027(4)	20.013(1)	19.947(4)	19.152(1)
β (°)					98.75(1)
U (Å³)	3604(2)	3581(2)	3553(2)	3523(2)	3061.4(15)
Z	4	4	4	4	4
D_c (g cm⁻³)	1.416	1.311	1.468	1.495	1.564
2θ range (deg)	3.0 to 55.0	3.0 to 55.0	3.0 to 55.0	3.0 to 55.0	3.0 to 55.0
μ (mm⁻¹)	2.634	2.640	3.381	3.830	4.362
F(000)	1540	1448	1564	1576	1416
No. of independent reflections	3564	4092	7373	4494	6019
No. of observed reflections	2718	2760	5970	3526	2678
No. of parameters refined	299	409	299	305	265
Data-to-parameters ratio	9.1:1	6.7:1	20:1	11.6:1	10.1:1
Goodness of fit	1.29	1.51	1.98	1.22	1.41
R_f (%)	4.02	4.61	4.75	3.1	6.18
R_w (%)	5.00	5.19	6.02	4.94	7.75
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å⁻³)	0.60, −0.40	0.43, −0.57	1.52, −1.38	0.75, −0.97	1.25, −0.96
Largest, mean Δ/σ	0.085, 0.010	0.034, 0.004	0.001, 0.000	0.002, 0.000	0.000, 0.000

Fig. 1. Perspective view of the molecular structure of 4. All hydrogen atoms are omitted for clarity. The thermal ellipsoids are drawn at the 35% probability level.
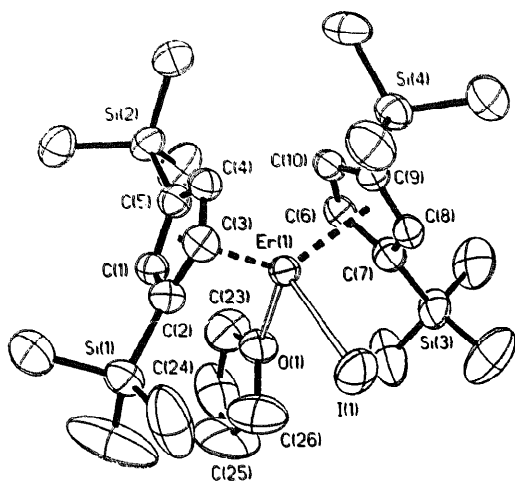

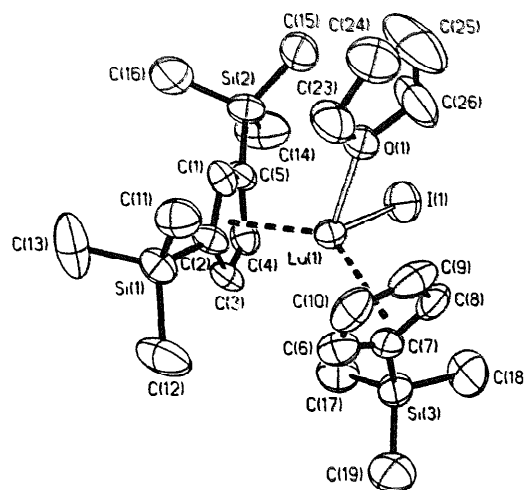Fig. 2. Perspective view of the molecular structure of 6. All hydrogen atoms are omitted for clarity. The thermal ellipsoids are drawn at the 35% probability level.

replacing one of the Cp″ by Cp′, 6 has a lower symmetry and crystallizes in space group $P2_1/c$ with $Z = 4$.

Fig. 1 represents the typical molecular structure of complexes 2–5. The molecular structure of complex 6 is shown in Fig. 2. A feature that is common to all five complexes is that each complex has an atomic arrangement typical of a bent metallocene complex with two additional ligands. As shown in Figs. 1 and 2, the two ring centroids of the cyclopentadienyl ligand plus the iodide and oxygen atom of the THF describe a distorted tetrahedral geometry around the lanthanide metal. The similarity of these structures is indicated by examining the compilation of values of the four angles (ring centroid)–metal–(ring centroid), (ring centroid)–metal–oxygen, (ring centroid)–metal–iodide and iodide–metal–oxygen given in Table 2. The (ring centroid)–metal–(ring centroid) angles fall in the range normally observed for bis(trimethylsilyl)cyclopentadienyl lanthanide complexes [2,15,17–19].

Table 2 lists the important bond distances and angles for 2–6. Comparing the mean Ln–Cent (ring centroid of the cyclopentadienyl), the mean Ln–C(ring) and Ln–O distances, we find that the decreases of the above values in going from 2–5 are those expected from the decrease in the eight-coordinate metal ionic radii [20], suggesting

the ionic interactions between metal and C_5 ring or oxygen atom of THF. For instance, a change of yttrium for samarium makes the metal–O and the average metal–C distances shorter by 0.061 and 0.060 Å respectively, consistent with the 0.06 Å difference in the ionic radii of yttrium and samarium [20]. The variations in the metal–iodide distances are, however, not particularly predictable. The Y–I distance differs from the Sm–I distance by 0.038 Å, which is significantly smaller than the 0.06 Å difference in the metal ionic radii. The Er–I distance is 0.038 Å shorter than that of Y–I, which is much greater than the 0.015 Å difference in their ionic radii. The difference between Er–I and Lu–I is 0.035 Å, close to the 0.027 Å difference in their ionic radii. These results suggest that some covalent bonding may be present in such metal–iodide bonds, presumably due to both the polarizing power of the Ln^{3+} cation and the more polarizable I^- anion.

3. Conclusion

On changing from the chloro [15,17] to the iodo ligand, the molecular structure of the complex shifts

Table 2
Selected bond distances (Å) and angles (°) for Cp″$_2$LnI(THF) and (Cp″)Cp′LuI(THF)

	Sm (2)	Y (3)	Er (4)	Lu (5)	(Cp″)Cp′LuI(THF) (6)
Ln–I	3.007(1)	2.969(1)	2.931(1)	2.896(1)	2.914(2)
Ln–O	2.411(9)	2.350(4)	2.335(7)	2.307(7)	2.310(10)
av. Ln–Cent	2.437	2.369	2.350	2.323	2.315
av. Ln–C	2.714(10)	2.654(4)	2.640(9)	2.611(10)	2.605(18)
Cent1–Ln–Cent2	129.4	130.4	130.5	130.7	131.3
Cent–Ln–I	110.3, 105.5	109.9, 105.2	110.0, 105.1	110.0, 105.2	108.2, 106.3
Cent–Ln–O	107.0, 104.9	106.3, 104.9	106.3, 104.8	105.8, 105.1	108.1, 103.0
O–Ln–I	94.0(2)	94.2(1)	94.1(2)	93.8(2)	93.5(3)

Cent: the ring centroid of cyclopentadienyl.

from the formally eight-coordinate dimer with the doubly bridging Ln–Cl units to the formally eight-coordinate monomer with terminal Ln–I bond because of the steric effect. This systematic study shows that the variations in the metal–iodide bond distances are not as predictable as those in metal–carbon and metal–oxygen bond distances, and the halide exchange reaction should be a convenient method to prepare organolanthanide iodide complexes.

4. Experimental section

4.1. General procedure

All experiments were performed under dry dinitrogen using standard Schlenk techniques, or in a glovebox. All solvents were distilled from sodium benzophenone ketyl immediately prior to use. Anhydrous lanthanide chlorides were prepared from the hydrates by standard procedures [21]. Cp″Na and Cp′Na were prepared according to the literature methods, but using NaH instead of KH [22,23]. [Cp″₂LnCl]₂ were prepared by the literature methods [15–17]. NaI was purchased from Aldrich Chemical Company and dried under vacuum at 200 °C. All other chemicals were purchased from Aldrich Chemical Company and used as received. Infrared spectra were obtained on a Perkin–Elmer 1600 Fourier transform spectrometer. Mass spectra were recorded on a HP5989A spectrometer. ¹H NMR spectra were recorded on a Bruker WM-250 spectrometer. Chemical shifts were assigned relative to C₆D₅H, 7.15 ppm.

4.2. Preparation of Cp″₂LaI(THF) (1)

A THF solution of Cp″Na (12.0 ml, 6.84 mmol) was slowly added to the stirring solution of LaCl₃ (0.85 g, 3.46 mmol) in 20 ml of THF at room temperature. The white suspension was stirred at room temperature for 24 h and then filtered. To the resulting clear solution ([Cp″₂LaCl]₂ [15–17]) was added anhydrous NaI (2.0 g, 13.3 mmol), and the reaction mixture was stirred overnight. The white precipitates (NaI + NaCl) were filtered off. After removal of THF, the residue was extracted with n-hexane three times (3 × 30 ml). The combined organic solution was then concentrated to give colorless crystallines which were filtered off and dried under vacuum (1.18 g, 50%). Anal. Found: C, 38.63; H, 6.44. C₂₂H₄₂Si₄LaI Calc.: C, 38.59; H, 6.19%. ¹H NMR: δ_H (C₆D₆) 6.85 (6H, br, m), 0.39 (36H, s). IR (KBr, cm⁻¹): 3065w (CH of Cp″), 2955s and 2899m (CH₃), 1250s and 832vs (Cp″). MS (EI): m/z (%) 669 (5) [Cp″₂LaI–CH₃]⁺, 557 (100) [Cp″₂La]⁺. Recrystallization from THF/hexane gave colorless crystals which unfortunately do not diffract.

4.3. Preparation of Cp″₂SmI(THF) (2)

This compound can be prepared either in an analogous manner to 1 or by an alternative method described below in 60% and 69% yields, respectively.

Alternate method. To a mixture of SmCl₃ (1.50 g, 5.84 mmol) and NaI (2.0 g, 13.3 mmol) in 40 ml of THF was slowly added the THF solution of Cp″Na (21.0 ml, 11.60 mmol) at room temperature. The reaction mixture was stirred at room temperature for 24 h and then filtered. After removal of THF, the residue was extracted with n-hexane three times (3 × 40 ml). The combined solution was then concentrated to give yellow crystallines which were filtered off and dried under vacuum (2.80 g, 69%). Anal. Found: C, 38.14; H, 6.23. C₂₂H₄₂Si₄SmI Calc.: C, 37.95, H, 6.08%. IR (KBr, cm⁻¹): 3060w (CH of Cp″), 2953s and 2899m (CH₃), 1251s and 835vs (Cp′). MS (EI): m/z (%) 682 (2) [Cp″₂SmI–CH₃]⁺, 570 (100) [Cp″₂Sm]⁺, 361 (9) [¹⁴²SmCp″]⁺. Recrystallization from THF/hexane gave yellow crystals which were identified to be 2 by X-ray analysis.

4.4. Preparation of Cp″₂YI(THF) (3)

This compound was prepared in an analogous manner to 1 except the crystals were suitable for X-ray diffraction without further recrystallization from THF/hexane, and isolated in 60% yield. Anal. Found: C, 45.86; H, 7.72. C₂₆H₅₄O₁.₅Si₄YI Calc.: C, 45.27; H, 7.33%. ¹H NMR: δ_H (C₆D₆) 6.83 (6H, br, m), 3.62 (6H, m), 1.17 (6H, m), 0.39 (36H, s). IR (KBr, cm⁻¹): 3066w (CH of Cp″), 2956s and 2899m (CH₃), 1251s and 834vs (Cp″), 1081s (THF). MS (EI): m/z (%) 619 (5) [M – (THF + CH₃)]⁺, 507 (90) [YCp″₂]⁺, 298 (5) [YCp″]⁺, 72 (67) [THF]⁺.

4.5. Preparation of Cp″₂ErI(THF) (4)

Method as for 3 using ErCl₃ (1.0 g, 3.65 mmol). The product formed pink crystallines from hexane. Recrystallization from THF/hexane gave shiny pink crystals (1.70 g, 59%). Anal. Found: C, 39.68; H, 6.52. C₂₆H₅₀OSi₄ErI Calc.: C, 39.78; H, 6.42%. IR (KBr, cm⁻¹): 3062w (CH of Cp″), 2954s and 2894m (CH₃), 1249s and 832vs (Cp″), 1080s (THF). MS (EI): m/z (%) 584 (22) [¹⁶⁶ErCp″₂]⁺, 72 (100) [THF]⁺.

4.6. Preparation of Cp″₂LuI(THF) (5)

This compound was prepared in an analogous manner to 3 and isolated in 65% yield. Anal. Found: C, 39.01; H, 6.50. C₂₆H₅₀OSi₄LuI Calc.: C, 39.38; H, 6.36%. ¹H NMR: δ_H (C₆D₆) 6.85 (6H, br, m), 3.63 (4H, m), 1.21 (4H, m), 0.46 (36H, s). IR (KBr, cm⁻¹):

3075w (CH of Cp"), 2956s and 2898 m (CH$_3$), 1250s and 835vs (Cp"), 1084s (THF). MS (EI): m/z (%) 720 (6) [CP"$_2$LuI]$^+$, 705 (15) [Cp"$_2$LuI–CH$_3$]$^+$, 593 (100) [Cp"$_2$Lu]$^+$, 384 (2) [^{175}LuCp"]$^+$, 72 (85) [THF]$^+$. Recrystallization from THF/hexane gave colorless crystals, Cp"$_2$LuI(THF) (5).

4.7. Preparation of (Cp")Cp'LuI(THF) (6)

A THF solution of Cp"Na (3.10 ml, 1.67 mmol) was slowly added to the stirring solution of LuCl$_3$ (0.47 g, 1.67 mmol) in 20 ml of THF at 0 °C. The reaction mixture was stirred overnight at room temperature. To the above solution was slowly added Cp'Na (5.60 ml, 1.67 mmol). The mixture was allowed to stir overnight at room temperature, followed by the addition of NaI (1.50 g, 10.0 mmol). The following work-up procedures were similar to the samarium analogue above. A white powder was isolated (0.23 g, 21%). Anal. Found: C, 34.31; H, 5.34. C$_{19}$H$_{34}$Si$_2$LuI Calc.: C, 35.18; H, 5.28%. ^{1}H NMR: δ_H (C$_6$D$_6$) 6.73–6.44 (7H, br, m), 0.25 (9H, s), 0.43 (18H, s). IR (KBr, cm^{-1}): 3081w (CH of Cp"), 2955s and 2897m (CH$_3$), 1249s and 837vs (Cp"). MS (EI): m/z (%) 633 (5) [(Cp")Cp'LuI–CH$_3$]$^+$, 521 (74) [(Cp")Cp'Lu]$^+$. Recrystallization from THF/hexane gave colorless crystals that were identified to be 6 by X-ray analysis.

4.8. X-ray structure determination

All single crystals were sealed under N$_2$ and immersed in Paraton-N oil in a thin-walled glass capillary. Data were collected on a Rigaku AFC7R diffractometer for **2, 5** and **6**, and on an MSC/Rigaku RAXIS-IIC imaging plate for **3** and **4** using Mo Kα radiation from a Rigaku rotating-anode X-ray generator operating at 50 kV and 90 mA. Absorption correction was applied by correlation of symmetry-equivalent reflections using the ABSCOR program [24], or using an empirical ψ-scan method. All five structures were solved by direct methods and subsequent Fourier difference techniques, and refined anisotropically for all non-hydrogen atoms by full-matrix least-squares on F using the Siemens SHELXTL PLUS program package (PC version) [25]. All hydrogen atoms were geometrically fixed using the riding model. Crystal data and details of data collection and structure refinement are given in Table 1.

5. Supplementary material

Tables of further details of the X-ray crystal structure determinations, complete atomic coordinates, thermal parameters, bond lengths and angles and atom-number-

ing schemes for **2–6**. Ordering information is given on any current masthead page.

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