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STUDIES ON ORGANOLANTHANIDE COMPLEXES

VIII *. SYNTHESIS AND THERMAL DECOMPOSITION OF NEW σ-BONDED 1,1′-TRIMETHYLENEDICYCLOPENTADIENYL-EARLY LANTHANIDE COMPLEXES

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Summary

1,1′-Trimethylenedicyclopentadienyl-early lanthanide chlorides, $[C_5H_4(CH_2)_3C_5H_4]LnCl \cdot THF$, in THF at −70°C reacted with aryllithium or alkyllithium, producing seven new THF solvated Ln–C σ-bonded 1,1′-trimethylenedicyclopentadienyl-early lanthanide complexes. The yttrium analogue was also synthesized. Their structures were determined by elemental analysis, infrared spectra, mass spectra, ^1H NMR and thermoanalyses. The thermal decomposition of the complexes obtained was studied at ambient temperature or 100°C.

Introduction

Studies on the formation and decomposition of cyclopentadienyl-organolanthanide complexes containing σ-bonds are of fundamental significance. Recently, the σ-bonded organo complexes with heavy lanthanide have been reported [1,2,3]. However, relatively few examples of cyclopentadienyl-early lanthanide complexes containing Ln–C σ-bonds have been studied [4,5,6]. Here we wish to report the syntheses of seven new σ-bonded tetrahydrofuran-solvated early lanthanide complexes, $[C_5H_4(CH_2)_3C_5H_4]LnAr \cdot THF$ and $[C_5H_4(CH_2)_3C_5H_4]LnR \cdot THF$ where $Ln = La$, Pr, Nd; $Ar = C_6H_5$, $p\text{-}CH_3C_6H_4$, $R = C(CH_3)_3$, $CH_2C(CH_3)_3$, by the reaction of 1,1′-trimethylenedicyclopentadienyl-early lanthanide chlorides in THF at −70°C with the corresponding aryllithium or alkyllithium, respectively. The yttrium analogue was also synthesized. The thermal decomposition of the tertiary butyl complexes **3**, **7** and **8** occurred at ambient temperature or 100°C.

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* For part VII see ref. 6.

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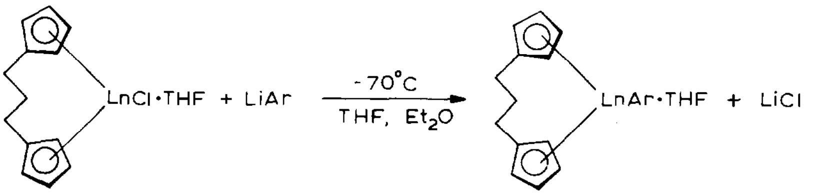
Results and discussion

On our previous publications we reported that when 1,1'-trimethylenedicyclopentadienyl instead of separated cyclopentadienyl was used as a ligand, the disproportionation of dicyclopentadienyl-early lanthanide chlorides was thus prevented, and the stabilization achieved [7,8]. This success lays the foundation for the syntheses of Ln–C σ-bonded 1,1'-trimethylenedicyclopentadienyl-early lanthanide complexes. 1,1'-Trimethylenedicyclopentadienyl-early lanthanide chlorides in THF at −70°C were allowed to react with aryllithium or alkyllithium, to afford THF solvated early lanthanide complexes containing the Ln–C σ-bond as depicted in eqs. 1 and 2, respectively.

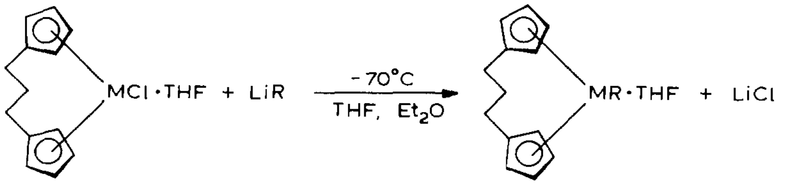
Tsutsui et al. synthesized analogous phenylethynyl complexes and claimed that these complexes were solvent free neutral ones [4]. However, all the products so far obtained were found to be THF solvated neutral complexes without exception. In order to stabilize early lanthanide complexes more ligands should be required to satisfy their coordination and stereo saturation. Obviously, the stabilities of solvated products should be better. These complexes containing a σ-bond are still quite sensitive to air and moisture, not able to sublime, and soluble in polar solvents, but slightly soluble in non-polar solvents. The physical properties and elemental analyses are listed in Table 1.

The infrared spectra of the aforementioned seven Ln–C σ-bonded early organolanthanides and the yttrium analogue were measured from 4000 to 200 cm^{-1}. All the characteristic absorption peaks due to cyclopentadienyl, coordinated THF, ring-



(1)

(1 : Ln = La, Ar = C_6H_5;
2: Ln = La, Ar = $C_6H_4CH_3$-*p*;
4: Ln = Pr, Ar = C_6H_5;
5: Ln = Pr, Ar = $C_6H_4CH_3$-*p*)



(2)

(3: M = La, R = $(CH_3)_3C$;
6: M = La, R = $(CH_3)_3CCH_2$;
7: M = Nd, R = $(CH_3)_3C$;
8: M = Y, R = $(CH_3)_3C$)

TABLE 1

σ-BONDED EARLY ORGANOLANTHANIDE AND ORGANOYTTRIUM COMPLEXES

Compound	Color	M.p. (°C)	Yield (%)	Analysis (Found(calcd.)(%))		
				Ln	C	H
$[C_5H_4(CH_2)_3C_5H_4]LaC_6H_5 \cdot THF$	Yellow	150–156 [a]	53	29.68	61.67	5.99
(1)				(30.30)	(60.26)	(5.94)
$[C_5H_4(CH_2)_3C_5H_4]LaC_6H_4CH_3\text{-}p \cdot THF$	orange-	140–142	30	29.40	60.34	6.06
(2)	yellow			(29.41)	(61.02)	(6.19)
$[C_5H_4(CH_2)_3C_5H_4]LaC(CH_3)_3 \cdot THF$	pale-	164–168 [a]	30	31.95	56.81	6.41
(3)	yellow			(31.69)	(57.53)	(7.13)
$[C_5H_4(CH_2)_3C_5H_4]PrC_6H_5 \cdot THF$	pale-	145–154 [a]	62	30.06	60.64	5.91
(4)	yellow			(30.61)	(60.00)	(5.91)
$[C_5H_4(CH_2)_3C_5H_4]PrC_6H_4CH_3\text{-}p \cdot THF$	pale-	115–116	73	29.44	59.86	6.27
(5)	yellow			(29.72)	(60.74)	(6.16)
$[C_5H_4(CH_2)_3C_5H_4]LaCH_2C(CH_3)_3 \cdot THF$	yellow	128–132	20	30.96	58.69	6.81
(6)				(30.70)	(58.41)	(7.35)
$[C_5H_4(CH_2)_3C_5H_4]NdC(CH_3)_3 \cdot THF$	pale-	144–147	26	32.95	56.93	6.46
(7)	green			(32.51)	(56.84)	(7.04)
$[C_5H_4(CH_2)_3C_5H_4]YC(CH_3)_3 \cdot THF$	yellow	172–178 [a]	70	22.97	64.09	7.98
(8)				(22.89)	(64.94)	(8.05)

[a] Decomposition temperature.

bridged methylene, aryl or alkyl are evidently present. Comparing the infrared spectra of σ-bonded organo complexes of early lanthanides with 1,1′-trimethylenedicyclopentadienyllanthanum chloride, all the four characteristic absorption peaks of a cyclopentadienyl shift to lower frequencies, e.g. 770, 1021 and 3070 cm^{-1} to 752, 1015 and 3060 cm^{-1}, respectively, probably because the electronegativity on the metal decreases as a result of the replacement of chlorine in 1,1′-trimethylenedicyclopentadienyllanthanum chloride by alkyl. It is worth noting that the stretching vibrations of =CH in aryl and cyclopentadienyl at 3060 cm^{-1} overlap each other, so the intensity of this peak becomes stronger. The characteristic absorption peaks of methylene also shift to lower frequencies and those of phenyl are assigned as follows: the peaks near 1500, 1598 and 1660 cm^{-1} are assigned to the in-plane vibration of the C=C skeleton, the peak at 700 cm^{-1} is assigned to the out-of-plane bending vibration of C–H on the monosubstituted phenyl ring. The band at 810–820 cm^{-1} is assigned to the out-of-plane bending vibration of C–H on the *para*-disubstituted phenyl ring, bands at 2950 and 2860 cm^{-1} to the C–H stretching vibration in t-butyl, the band at 1458 cm^{-1} to the C–H twisting vibration in $C\text{–}CH_3$ and the bands at 1250 and 1200 cm^{-1} to the $C(CH_3)_3$ skeleton vibration. In the low frequency region, we found that when the chlorine in 1,1′-trimethylenedicyclopentadienyllanthanum chloride was replaced by alkyl, a 200 cm^{-1} band disappeared.

Mass spectra of **2** did not show a parent molecular ion, but trimethylene, cyclopentadienyl, tetrahydrofuran, tolyl, 1,1′-trimethylenedicyclopentadienyl ions and fragments which were attributed to the moieties containing the above-mentioned groups connected with the metal appeared clearly.

Thermoanalysis of complex **1** was carried out from 70–300°C in an atmosphere of nitrogen and the weight loss was equal to 15.28% which was consistent with the loss of one mole of tetrahydrofuran.

TABLE 2

NMR PROTON SHIFTS (δ, TMS (ppm))

Compound	Ring protons	Methylene protons	THF protons	Aryl or alkyl protons
$[C_5H_4(CH_2)_3C_5H_4]LaC_6H_5 \cdot THF$	5.86(m), 5.71(m)	2.43(m)	3.50(t), 1.68(m)	7.21(m)
$[C_5H_4(CH_2)_3C_5H_4]LaC_6H_4CH_3\text{-}p \cdot THF$	5.82(m), 5.65(m)	2.38(m)	3.46(t), 1.62(m)	7.37(m), 2.22(s)
$[C_5H_4(CH_2)_3C_5H_4)LaC(CH_3)_3 \cdot THF$	5.76(m), 5.60(m)	2.33(m)	3.43(t), 1.57(m)	1.12(s)
$[C_5H_4(CH_2)_3C_5H_4]LaCH_2C(CH_3)_3 \cdot THF$	5.82(m), 5.68(m)	2.43(m)	3.54(t), 1.68(m)	1.21(s), 0.81(s)
$[C_5H_4(CH_2)_3C_5H_4)YC(CH_3)_3 \cdot THF$	5.66(bm)	2.46(m)	3.54(m), 1.65(m)	1.17(s)

^{1}H NMR spectra of **1**, **2**, **3**, **6** and **8** were measured using perdeuterotetrahydrofuran as solvent. The results are listed in Table 2. ^{1}H NMR spectra indicate that the presence of coordinated tetrahydrofuran and its chemical shift is different from that of free THF as a result of THF coordinating with the rare earth metal. Because of the coupling of yttrium with the protons on cyclopentadienyl, the ^{1}H NMR signal due to ring hydrogens of complex **8** becomes broad due to the overlap of two independent multiplets.

Contrary to *d*-transition metal chemistry, the organolanthanide complex is more stable toward β-hydrogen elimination. So complexes having β-hydrogen atoms can also be synthesized. For example, complex **3**, $[C_5H_4(CH_2)_3C_5H_4]LaC(CH_3)_3 \cdot THF$ decomposes in the solid state at 100°C over 4 h in vacuum to yield 2-methylpropane and 2-methylpropene in the ratio 1.2/1, similar to the β-hydrogen elimination products of dicyclopentadienyl-t-butyllutetium [3]. Besides 2-methylpropane and 2-methylpropene, ^{1}H NMR spectra indicated the existence of one other decomposition product, which is a liquid at room temperature, and seems to be tetrahydrofuran. The complex **8**, too, decomposed to generate 2-methylpropane and 2-methylpropene at ambient temperature. Table 3 shows that the decomposition of complex **8** is fast at the beginning, but slows down towards the end. The ratio of 2-methylpropane to 2-methylpropene in the decomposition product is dependent on the temperature. At 100°C the ratio is near 1/1 for complex **3**, but at ambient temperature it changes to 4/1 (Table 4). Furthermore, at ambient temperature the ratio for complex **8** is within the range 4/1 to 6/1. If the thermal decomposition proceeds only via β-hydrogen elimination, an equivalent quantity of 2-methylpropane and 2-methylpropene should be produced. In the case of complex **8** decomposing at ambient temperature the quantity of 2-methylpropane is much more than that of 2-methylpropene, because 2-methylpropene, the β-hydrogen elimination product, probably abstracts the hydrogen atom from tetrahydrofuran to give a high yield of 2-methylpropane [9]. The absence of equivalent volumes of 2-methylpropane and 2-methylpropene in this decomposition is remarkable and suggests that β-hydrogen elimination is not the most facile decomposition pathway and also implies unusual reactivity patterns for organo-rare earth complexes [9]. Evans reported that $(C_5H_5)_2YC(CH_3)_3 \cdot THF$ decomposed at ambient temperature overnight [10]. One

TABLE 3

$[C_5H_4(CH_2)_3C_5H_4]YC(CH_3)_3 \cdot THF$ THERMAL DECOMPOSITION

Time [a] (h)	Volume of generated gas (10^{-3} ml/mmol)		$CH_3(CH_3)CHCH_3/CH_3(CH_3)C{=}CH_2$
	$CH_3(CH_3)CHCH_3$	$CH_3(CH_3)C{=}CH_2$	
1	32	8.3	4/1
2	42	11	4/1
3	43	11	4/1
6	46	9	5/1
12	69	13	5/1
24	71	14	5/1
48	96	16	6/1
Three months	1000	190	5/1

[a] A sample was kept for a period of time at ambient temperature.

TABLE 4

THE EFFECTS OF METALS AND LIGANDS ON DECOMPOSITION

Compound	Volume of generated gas [a] (10^{-4} ml/mmol)		$CH_3(CH_3)CHCH_3$/ $CH_3(CH_3)C{=}CH_2$
	$CH_3(CH_3)CHCH_3$	$CH_3(CH_3)C{=}CH_2$	
$[C_5H_4(CH_2)_3C_5H_4]YC(CH_3)_3 \cdot THF$	460	90	5/1
$[C_5H_4(CH_2)_3C_5H_4]LaC(CH_3)_3 \cdot THF$	170	41	4/1
$[C_5H_4(CH_2)_3C_5H_4]NdC(CH_3)_3 \cdot THF$	97	18	5/1
$(C_5H_5)_2LaC(CH_3)_3(phen)_2$ [b,c]	7	1.3	5/1
$(C_5H_5)_2NdC(CH_3)_3(phen)$ [b,c]	55	14	4/1

[a] An open ampoule containing the sample, was evacuated for 1 h, sealed and kept at ambient temperature for 6 h. Then the ampoule was reopened, and the gas released was identified and determined by gas chromatography. [b] Ref. 6. [c] phen = *o*-phenanthroline.

mmol of complex **8** released 1.0 ml of 2-methylpropane and 0.19 ml of 2-methylpropene, after the complex had been kept for 3 months at ambient temperature in an ampoule filled with argon. This fact indicates that complex **8** decomposed quite slowly in the ampoule, with only 4.5% decomposition after 3 months, and was stable enough to be isolated. The effects of metals and ligands on decomposition are listed in Table 4.

More gas was released in the case of complexes containing 1,1′-trimethylenedicyclopentadienyl and tetrahydrofuran than those complexes containing *o*-phenanthroline and cyclopentadienyl no matter what metal, lanthanum or neodymium, was present. The coordination number and stereo saturation of the complexes in which cyclopentadienyl and the bulky *o*-phenanthroline were used as ligands become higher than that of the corresponding complexes containing 1,1′-trimethylenedicyclopentadienyl ligands. Therefore the complexes containing *o*-phenanthroline were more stable and decomposed only to a small extent. The volume of released gas for complexes containing 1,1′-trimethylenedicyclopentadienyl and tetrahydrofuran decreased in the order Y > La > Nd, whereas the volume of released gas for *o*-phenanthroline complexes increased in the order La < Nd. Since two molecules of *o*-phenanthroline are present in the dicyclopentadienyl-t-butyllanthanum complex, but only one molecule of *o*-phenanthroline in the corresponding neodymium complex, the former complex is stable towards decomposition.

Experimental

All operations on these organolanthanide complexes were performed under prepurified argon using Schlenk techniques or in a glovebox. All solvents were refluxed and distilled over either finely divided $LiAlH_4$ or blue sodium benzophenone under argon immediately before use. 1,1′-Trimethylenedicyclopentadienyllanthanide chlorides were prepared as previously described [7]. *p*-$CH_3C_6H_4Li$ and C_6H_5Li were prepared by the method of Gilman et al. [11], t-BuLi [12] and $(CH_3)_3CCH_2Li$ [13] were prepared according to published procedures. Melting points and thermal decomposition temperatures were determined in sealed argon filled capillaries and were uncorrected. Infrared spectra were recorded on Perkin-Elmer 983 or Digilab FTS-20/E Fourier Transform IR Spectrometers as

Nujol and Fluorolube mulls and were examined between disk-shaped CsI crystals or polyethylene plates; the mulls were prepared in an argon filled glovebox. Mass spectra and GC/MS were recorded on a Finnigan 4021 spectrometer. ^{1}H NMR spectra were obtained on an EM 360 L (60 MHz) spectrometer and referenced to external Me_4Si.

Analyses of rare earth metals were accomplished using a direct complexometric titration procedure with disodium EDTA [2]. Carbon and hydrogen analyses were measured by the combustion method. Sampling was carried out under argon and the sample analyzed was sealed in a small aluminum tube.

Thermal decomposition

Thermal decomposition was carried out as follows: 90 mg of 1,1′-trimethylenedicyclopentadienyl-t-butyllanthanum tetrahydrofuran complex was heated at 100°C in vacuo in a closed vessel for 4 h, the gas, released and trapped, was identified and determined by gas chromatography based on peak height. Moreover the gas was identified by GC/MS. The procedure for the thermal decomposition study at ambient temperature is as follows: an ampoule containing an exactly weighed amount of complex on a 0.1 mmol scale was opened, then the ampoule was evacuated for 1 h in order to exhaust the gas, which had been released before, the ampoule was then sealed, and deposited at ambient temperature for a definite time. The ampoule was again opened, the gas released was identified and determined by gas chromatography based on peak height. The gas was also identified by GC/MS.

1,1′-Trimethylenedicyclopentadienyl(phenyl)lanthanum · THF (1)

Anhydrous $LaCl_3$ (21.1 mmol) was treated with $[C_5H_4(CH_2)_3C_5H_4]Na_2$ to give 12.6 mmol of 1,1′-trimethylenedicyclopentadienyllanthanum chloride according to the procedure previously described [7]. An equivalent phenyllithium ether solution was added slowly to the resulting 1,1′-trimethylenedicyclopentadienyllanthanum chloride-THF solution at −70°C. The reaction mixture was then allowed to warm to ambient temperature in 2 h, and stirred for an additional 18 h. After removal of the solvent the resulting brown oil was extracted with 120 ml of benzene. The Schlenk flask was centrifuged, the solid was removed from the solution, and the benzene solution was concentrated to give a solid. The resulting solid was extracted again with 110 ml of benzene. After isolation of the solid, the benzene was removed in vacuo to give a solid product. The solid product was washed with 20 ml of hexane then dried in vacuo to give a yellow solid. Yield 53%; decomposition temperature 150–156°C. Anal. Found: C, 61.67; H, 5.99; La, 29.68. $C_{23}H_{27}LaO$ calcd.: C, 60.26; H, 5.94; La, 30.30%. IR (cm^{-1}): 226s, 245m, 299w, 340s, 385s, 540s, 615w, 675m, 700m, 752vs, 812s, 891m, 918w, 1015s, 1032s, 1045s, 1259w, 1445s, 1485s, 1595m, 1652s, 2848s, 2919s, 3053s.

1,1′-Trimethylenedicyclopentadienyl(tolyl)lanthanum · THF (2)

The synthesis of 1,1′-trimethylenedicyclopentadienyltolyllanthanum-tetrahydrofuran was similar to that of **1**. IR (cm^{-1}): 230s, 245m, 299w, 340s, 385s, 540s, 676m, 708w, 754vs, 810m, 893m, 921w, 1017s, 1032s, 1044m, 1442s, 1485m, 1595m, 1655m, 2850s, 2920vs, 3060s.

1,1′-Trimethylenedicyclopentadienyl(phenyl)praseodymium · THF (4)

The synthesis of 1,1′-trimethylenedicyclopentadienylphenylpraseodymium-tetrahydrofuran was similar to that of **1**. IR (cm^{-1}): 235m, 350s, 435s, 676m, 700w, 755s, 812w, 892m, 900s, 1014s, 1030s, 1045m, 1240m, 1435s, 1485m, 1600s, 1668s, 2860vs, 2924vs, 3060s.

*1,1'-Trimethylenedicyclopentadienyl(tolyl)praseodymium · THF (**5**)*

The synthesis of 1,1'-trimethylenedicyclopentadienyltolylpraseodymium-tetrahydrofuran was similar to that of **1**. IR (cm^{-1}): 222s, 245m, 299w, 350s, 385s, 550s, 664m, 675m, 755s, 800m, 820m, 864m, 890m, 921w, 1021s, 1034s, 1045s, 1250m, 1437s, 1500m, 1600m, 1660m, 2849vs, 2919vs, 3063s.

*1,1'-Trimethylenedicyclopentadienyl(t-butyl)lanthanum · THF (**3**)*

Anhydrous $LaCl_3$ (5.07 g, 20.7 mmol) was allowed to react with $[C_5H_4(CH_2)_3C_5H_4]Na_2$ to give 15 mmol of 1,1'-trimethylenedicyclopentadienyllanthanum chloride according to the procedure previously described [7]. An equivalent t-butyllithium ether solution was added slowly to the resulting 1,1'-trimethylenedicyclopentadienyl chloride THF solution at −70°C. Then the reaction mixture was allowed to warm to ambient temperature over 2 h, and then stirred for another 18 h. The solvent was removed in vacuo and the residue was extracted with 120 ml of toluene. After isolation of the solid, removal of the toluene gave a viscous solid, which was extracted again with 120 ml of toluene, the residue was removed and the toluene solution was concentrated in vacuo to give a light yellow solid of $[C_5H_4(CH_2)_3C_5H_4]LaC(CH_3)_3 \cdot THF$ which was washed with hexane and dried under vacuum. Yield 30%; decomposition temperature 164–168°C. Anal. Found: C, 56.81; H, 6.41; La, 31.95. $C_{21}H_{31}LaO$ calcd.: C, 57.53; H, 7.13; La, 31.69%. IR (cm^{-1}): 235m, 330s, 470s, 750vs, 810w, 894m, 900m, 1015m, 1030s, 1047s, 1440s, 1458s, 1605w, 1670m, 2860s, 2930s, 2950s, 3060m.

*1,1'-Trimethylenedicyclopentadienyl(neopentyl)lanthanum · THF (**6**)*

$[C_5H_4(CH_2)_3C_5H_4]LaCH_2C(CH_3)_3 \cdot THF$ was synthesized in an analogous manner to that described for **3**. IR (cm^{-1}): 225s, 250m, 325m, 347m, 388s, 660w, 670w, 748s, 805m, 850w, 890w, 895w, 915w, 1015s, 1030s, 1040m, 1258m, 1438s, 1600m, 2850s, 2920s, 2950s, 3060m.

*1,1'-Trimethylenedicyclopentadienyl(t-butyl)neodymium · THF (**7**)*

$[C_5H_4(CH_2)_3C_5H_4]NdC(CH_3)_3 \cdot THF$ was synthesized in an analogous manner to that described for **3**. IR (cm^{-1}): 220w, 255w, 388s, 550m, 660w, 672m, 755s, 810s, 850w, 895m, 1020s, 1030s, 1040m, 1195w, 1256m, 1438s, 1450m, 1595m, 2850s, 2920s, 2940s, 3060m.

*1,1'-Trimethylenedicyclopentadienyl(t-butyl)yttrium · THF (**8**)*

$[C_5H_4(CH_2)_3C_5H_4]YC(CH_3)_3 \cdot THF$ was synthesized in an analogous manner to that described for **3**. IR (cm^{-1}): 225w, 295m, 323m, 388s, 670w, 760s, 815s, 850w, 890m, 894m, 1015s, 1030s, 1043m, 1200w, 1258w, 1436s, 1450m, 1598m, 2845s, 2916s, 2940s, 3058m.

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