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Synthesis and crystal structure of $[(2,4\text{-}C_7H_{11})_2LnC{\equiv}CC_6H_5]_2$ (Ln = Gd, Er)

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Abstract

The reaction of $(2,4\text{-}C_7H_{11})_3Ln$ (Ln = Gd, Er) with $HC{\equiv}CC_6H_5$ in toluene leads to $[(2,4\text{-}C_7H_{11})_2LnC{\equiv}CC_6H_5]_2$, which crystallizes in the space group *Pabc*. The molecule shows the dimer structure, where two $(2,4\text{-}C_7H_{11})_2Gd$ or Er units are connected by asymmetrical alkynide bridges. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Alkynide; Crystal structure; 2,4-Dimethylpentadienyl–lanthanide; Organolanthanide; Synthesis

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1. Introduction

Since 1980, pentadienyl–lanthanoid chemistry has been actively investigated, partly because the pentadienyl–lanthanoid complexes possess reasonable thermal stability as well as chemical and catalytic reactivities. For lanthanides, the anionic pentadienyl group can be expected to bond favorably to ionic lanthanide complexes. Indeed, a few pentadienyl–lanthanide complexes, $Ln(2,4\text{-}C_7H_{11})_3$, (Ln = Nd [1], Gd [2], Er [3]), $[Nd_6(2,4\text{-}C_7H_{11})_6Cl_{12}\ (THF)_2]$ [4] and $Sm(C_8H_8)(2,4\text{-}C_7H_{11})(THF)$ [5] have recently been synthesized and characterized by single crystal X-ray diffraction. However, in contrast to the cyclopentadienyl ligands, 'open ring' pentadienyl–lanthanide compounds are still quite scarce, and very little is known about their reactions. Thus, the reaction of cobaltous chloride with two equivalents of the 2,4-dimethylpentadienyl anion ($2,4\text{-}C_7H_{11}^-$) leads to a dimeric complex, in which each cobalt center is bonded to an η^5-2,4-C_7H_{11} ligand and an η^4-diene portion of the 2,4,7,9-tetramethyl-2,4,6,8-decatetraene bridging unit, which was formed from the coupling of two pentadienyl units [6]. From the metathesis of (2,4-dimethylpentadienyl–potassium with lutetium trichloride (3:1 reaction stoichiometry) has been isolated an unusual minor reaction product, $[\eta^5\text{–}(CH_3)_2C_5H_5]Lu[\eta^5,\eta^3\text{-}(CH_3)C_5H_5CH_2CH_2CH(CH_3)C_3H_3(CH_3)]$, which contains a dimeric chelate ligand derived from the end-to-end fusion of two 2,4-dimethylpentadienyl groups [7]. As part of our investigation of the reactivity of pentadienyl–lanthanide compounds, we have examined the reaction of tris–(2,4-dimethylpentadienyl) lanthanide with phenylacetylene. This method is different from the others, such as the reaction between lanthanide halides and alkali metal acetylides [8–10], and the reaction of lanthanide alkyls [11,12] or divalent lanthanocene [13–15] with terminal alkyne. We report here the synthesis and X-ray structure of a novel complex $[(2,4\text{-}C_7H_{11})_2LnC{\equiv}CC_6H_5]_2$ (Ln = Gd,Er).

2. Experimental

All manipulations were conducted under dry nitrogen using the Schlenk techniques. The THF was predried with NaOH and distilled from sodium benzophenone ketyl. Anhydrous $GdCl_3$ was prepared by Taylor's method [16]. The complex $(2,4\text{-}C_7H_{11})_3Ln$ (Ln = Gd or Er) was prepared by a procedure published previously [2].

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Metal analysis was carried out by complexometric titration.

2.1. *Synthesis of $[(2,4\text{-}C_7H_{11})_2GdC{\equiv}CC_5H_6]_2$*

$PhC{\equiv}CH$ (0.2 g, 1.96 mmol) and $(2,4\text{-}C_7H_{11})_3Gd$ (0.85 g, 1.92 mmol) were placed in a Schlenk flask, and 10 ml of toluene was added. The mixture was stirred at 60°C for 2 h. The solution was concentrated and crystallized at -20°C to give red crystals, 0.55g (yield 60%). M.p. 167°C. Found: C, 58.41; H, 5.84; Gd, 34.65. Anal. Calc. $C_{22}H_{27}Gd$: C, 58.88; H, 6.0; Gd, 35.03%. The IR absorption peeks of the Gd complex (cm^{-1}): 3086 m, 2963 m, 2918 m, 2037 m, 1522 s, 1486 m, 1459 s, 1418 m, 1386 s, 1262 w, 1190 w, 1052 w, 1023 w, 848 m, 789 s, 692 s, 623 m, 561 m, 548 m, 493 m, 466 m. The synthesis of the Er analog is the same as above. The Er complex is red, yield 55%. Found: Er 36.15. Anal. Calc. 36.32. Er complex (cm^{-1}): 3082 m, 2962 m, 2923 m, 2037 s, 1513 s, 1468 s, 1443 m, 1382 s, 1262 w, 1190 w, 1070 w, 1023 m, 848 m, 793 m, 757 s, 691 s, 623 m, 530 m, 514 m, 493 m, 434 m. The MS main peaks for Gd complex (m/z): $[2M-C_7H_{11}]^+$ 793(29), $[2M-C_8H_5]^+$ 787(77), $[2M-C_7H_{11}-C_8H_5]^+$ 692(2), M^+444(8), $[M-C_7H_{11}]^+$ 349(2), $[M-C_8H_{11}]^+$ 343(9), $[C_8H_5]^+$ 101(3), $[C_7H_{11}]^+$ 95(39).

2.2. *Determination of crystal structure*

A crystal was sealed in a thin-walled glass capillary under nitrogen. The unit cell parameters were determined and X-ray intensity data were collected on a Nicolet $R_{3m/E}$ four-circle diffractometer, using graphite monochromated Mo–K_α radiation ($\lambda = 0.71069$ Å) in the ω scan mode. A total of 5896 (4325) reflections were collected within $3 < 2\theta < 56°$ (Gd) [$2 < 2\theta < 50°$(Er)]. The intensity of one check reflection was monitored every 68 reflections. No obvious change was observed in the intensities of the check reflection. Correction was made for Lorenz and polarization effects. The crystal data are listed in Table 1.

Calculations were carried out with the SHELXTL computer program. The position of the Gd (Er) atom was determined from a three-dimensional Patterson map followed by the use of the Fourier technique. The positions of all the non-hydrogen atoms could be fixed on different Fourier maps. They were refined by least squares and, at the final stage, refined anisotropically. The final agreement factors are $R = 0.045$ (0.0376) and $R_w = 0.046$ (0.0347).

Atomic coordinates and isotropic thermal parameters selected bond lengths, bond angles and plane equations are given in Tables 2–5, respectively.

Table 1
Crystal data of Gd and Er complexes

Compound	$[(2,4\text{-}C_7H_{11})_2GdC{\equiv}CC_6H_5]_2$	$[(2,4\text{-}C_7H_{11})_2ErC{\equiv}CC_6H_5]_2$
Molecular weight	897.5	916.4
Crystal size (mm)	$0.34\times0.28\times0.8$	$0.2\times0.32\times0.4$
Unit cell dimensions		
a (Å)	10.515(4)	10.473(2)
b (Å)	17.797(8)	20.912(4)
c (Å)	20.895(8)	17.813(5)
V (Å^3)	3910(2)	3901(1)
Scan range 2θ (°)	3–5θ	2–5θ
Reflections	5896	4325
Reflections for $I>3\sigma(I)$	2609	1839
Space group	*Pabc*	*Pabc*
Z	4	4
$D_{calc.}$ (f cm^{-3})	1052	1.56
R	0.045	0.038
R_w	0.046	0.035
$F(000)$	1784	1815

3. Results and discussion

3.1. *The syntheses of $(2,4\text{-}C_7H_{11})_2LnC{\equiv}CC_6H_5$ (Ln = Gd or Er)*

In general, the metathesis between the σ-bond alkyl–lanthanide and phenylacetylene is used to pre-

Table 2
Atomic coordinates (10^4) and isotropic thermal parameters (Å × 10^4) of Gd complex

	x	y	z	U^a
Gd	723(1)	5530(1)	5717(1)	42(1)
C(11)	2230(1)	6816(5)	3455(4)	61(4)
C(12)	3046(10)	7206(5)	3068(4)	68(4)
C(13)	4296(9)	6999(5)	3031(4)	68(4)
C(14)	4745(9)	6420(5)	3375(4)	67(4)
C(15)	3926(8)	6009(5)	3775(4)	58(3)
C(16)	2654(8)	6203(4)	3817(4)	47(3)
C(17)	1781(8)	5791(4)	4214(4)	46(3)
C(18)	1023(8)	5435(4)	4527(4)	48(3)
C(21)	3447(9)	5513(6)	5660(4)	83(4)
C(22)	3156(7)	5689(5)	6262(4)	62(3)
C(23)	2324(8)	5340(5)	6698(4)	60(3)
C(24)	1727(8)	4608(5)	6626(4)	57(3)
C(25)	1780(11)	4149(6)	6111(5)	91(5)
C(26)	860(11)	4383(6)	7166(6)	96(5)
C(27)	3718(10)	6421(6)	6522(6)	86(4)
C(31)	−853(11)	6092(6)	6668(5)	80(4)
C(32)	−1493(8)	6247(5)	6121(4)	55(3)
C(33)	−1023(7)	6614(5)	5546(4)	50(3)
C(34)	118(8)	7013(4)	5461(5)	56(3)
C(35)	1130(10)	7035(5)	5870(6)	81(4)
C(36)	241(10)	7417(5)	4844(5)	76(4)
C(37)	−2844(9)	5957(5)	6058(5)	70(4)

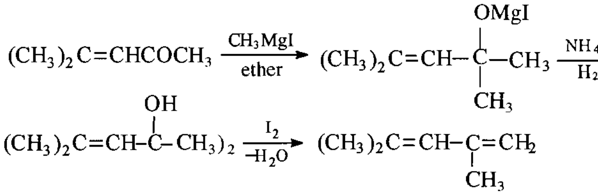
[a] Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor.

Table 3
Atomic coordinates (10^4) and isotropic thermal parameters (Å × 10^4) of Er complex

	x	*y*	*z*	*U*[a]
Er	721(1)	5717(1)	4469(1)	41(1)
C(11)	2245(10)	3460(5)	3199(5)	50(4)
C(12)	3042(10)	3060(5)	2805(6)	58(4)
C(13)	4308(11)	3022(5)	3001(6)	60(4)
C(14)	4736(10)	3375(6)	3583(6)	63(5)
C(15)	3926(8)	3771(5)	3985(5)	45(4)
C(16)	2659(9)	3822(4)	3799(5)	35(3)
C(17)	1779(8)	4209(4)	4209(5)	34(3)
C(18)	1021(8)	4528(4)	4568(5)	39(3)
C(21)	3438(8)	5666(5)	4499(6)	58(4)
C(22)	3162(9)	6261(5)	4305(6)	46(4)
C(23)	2320(9)	6692(5)	4680(5)	44(4)
C(24)	1710(9)	6646(5)	5383(5)	46(4)
C(25)	1761(10)	6135(5)	5847(6)	67(5)
C(26)	852(11)	7178(6)	5614(7)	76(5)
C(27)	3713(10)	6525(6)	3587(6)	68(5)
C(31)	−875(10)	6663(5)	3897(6)	62(4)
C(32)	−1497(9)	6117(5)	3751(5)	44(4)
C(33)	−1034(8)	5558(4)	3383(5)	38(3)
C(34)	139(10)	5455(5)	2980(5)	44(4)
C(35)	1115(9)	5876(5)	2955(5)	51(4)
C(36)	234(11)	4831(5)	2585(6)	62(4)
C(37)	−2862(10)	6051(6)	4045(6)	64(4)

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

pare the titled complexes. In this paper, the metathesis between the π-bond 2,4-dimethylpentadienyl and phenylacetylene was first adopted. The preparation of 2,4-dimethylpentadienyl ligand was as follows [17]:



$$\mathrm{(CH_3)_2C{=}CHCOCH_3 \xrightarrow[ether]{CH_3MgI} (CH_3)_2C{=}CH{-}\overset{OMgI}{\underset{CH_3}{C}}{-}CH_3 \xrightarrow[H_2O]{NH_4Cl}}$$
$$\mathrm{(CH_3)_2C{=}CH{-}\overset{OH}{C}{-}CH_3)_2 \xrightarrow[-H_2O]{I_2} (CH_3)_2C{=}CH{-}\underset{CH_3}{C}{=}CH_2} \quad (1)$$

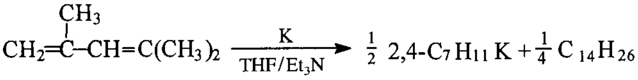
Table 4
Bond lengths (Å) of Gd and Er complexes

Gd–C(18)	2.512(8)	Gd–C(21)	2.866(10)
Gd–C(22)	2.814(8)	Gd–C(23)	2.673(8)
Gd–C(24)	2.723(9)	Gd–C(25)	2.820(10)
Gd–Gda	3.855(1)	C(11)–C(12)	1.370(13)
Gd–C(18a)	2.566(8)	C(16)–C(17)	1.438(11)
C(17)–C(18)	1.210(12)	C(18)–Gda	2.566(8)
C(21)–C(22)	1.332(13)	C(22)–C(23)	1.407(12)
C(22)–C(27)	1.531(14)	C(23)–C(24)	1.453(12)
C(24)–C(25)	1.352(14)	C(24)–C(26)	1.505(15)
Er–C(18)	2.512(9)	Er–C(21)	2.847(8)
Er–C(22)	2.813(9)	Er–C(23)	2.665(10)
Er–C(24)	2.738(10)	Er–C(25)	2.826(10)
Er–Era	3.856(1)	Er–C(18a)	2.557(9)
C(11)–C(12)	1.375(14)	C(16)–C(17)	1.428(12)
C(17)–C(18)	1.218(12)	C(18)–Era	2.557(9)
C(21)–C(22)	1.323(15)	C(22)–C(23)	1.427(14)
C(22)–C(27)	1.509(15)	C(23)–C(24)	1.409(13)
C(24)–C(25)	1.351(15)	C(24)–C(26)	1.488(15)

Table 5
Bond angles (Å) of Gd and Er complexes

C(18)–Gd–C(21)	80.4(3)	C(18)–Gd–C(22)	107.1(3)
C(21)–Gd–C(22)	27.1(3)	C(18)–Gd–C(23)	132.1(3)
C(21)–Gd–C(23)	53.2(3)	C(22)–Gd–C(23)	29.6(3)
C(18)–Gd–C(24)	126.9(3)	C(21)–Gd–C(24)	58.6(3)
C(22)–Gd–C(24)	55.0(3)	C(23)–Gd–C(24)	31.2(3)
C(18)–Gd–C(25)	100.4(3)	C(21)–Gd–C(25)	67.0(3)
C(22)–Gd–C(25)	67.1(3)	C(23)–Gd–C(25)	54.4(3)
C(18)–Gd–C(18a)	81.2(3)	C(15)–C(16)–C(17)	121.8(7)
C(16)–C(17)–C(18)	177.4(9)	Gd–C(18)–Gda	98.8(3)
C(17)–C(18)–Gda	135.6(7)	C(21)–C(22)–C(23)	130.5(9)
C(22)–C(23)–C(24)	126.7(8)	C(23)–C(24)–C(25)	127.3(8)
C(21)–Er–C(22)	27.0(3)	C(22)–Er–C(23)	30.0(3)
C(23)–Er–C(24)	30.2(3)	C(24)–Er–C(25)	28.0(3)
C(21)–Er–C(25)	66.9(3)	C(18)–Er–C(18a)	81.0(3)
Er–C(18)–Era	99.0(3)	Er–C(18)–C(17)	126.0(7)
Era–C(18)–C(17)	135.0(7)	Cent(1)–Er–Cent(2)	127.7
Cent(1)–Er–C(18)	112.0	Cent(1)–Er–C(18a)	114.8
Cent(2)–Er–C(18)	108.5	Cent(2)–Er–C(18a)	102.9
C(21)–C(22)–C(27)	127.3(9)	C(21)–C(22)–C(27)	118.9(9)
C(23)–C(22)–C(27)	113.7(9)	C(22)–C(23)–C(24)	130.7(9)
C(23)–C(24)–C(25)	125.3(9)	C(23)–C(24)–C(26)	117.9(9)
C(25)–C(24)–C(26)	116.6(9)	C(15)–C(16)–C(17)	120.0(8)
C(16)–C(17)–C(18)	178.7(9)		

The 2,4-dimethylpentadienyl potassium was prepared according to the literature [18]:



$$\mathrm{CH_2{=}\overset{CH_3}{C}{-}CH{=}C(CH_3)_2 \xrightarrow[THF/Et_3N]{K} \tfrac{1}{2}\ 2,4\text{-}C_7H_{11}K + \tfrac{1}{4}C_{14}H_{26}} \quad (2)$$

In order to inhibit the polymerization of 1,3-dimethylpentadiene triethylamine was added during the reaction process.

The reaction of $GdCl_3$ or $ErCl_3$ with three equivalents. of $(2,4\text{-}C_7H_{11})K$ leads to the formation of a crystalline compound, $(2,4\text{-}C_7H_{11})_3Gd$ or $(2,4\text{-}C_7H_{11})_3Er$, respectively which has been isolated and determined for its structure (Eq. (3)). It reacts with $HC{\equiv}CC_6H_5$ at 1:1 molar ratio in toluene to form $[(2,4\text{-}C_7H_{11})_2GdC{\equiv}CC_6H_5]_2$ or $[(2,4\text{-}C_7H_{11})_2ErC{\equiv}CC_6H_5]_2$ (Eq. (4)).

$$LnCl_3 + 3(2,4\text{-}C_7H_{11})K \overset{THF}{\rightarrow} (2,4\text{-}C_7H_{11})_3Ln + KCl \quad (3)$$

$$\begin{aligned}&(2,4\text{-}C_7H_{11})_3Ln\\&\quad + C_6H_5C{\equiv}CH \overset{Toluene}{\rightarrow} [(2,4\text{-}C_7H_{11})_2LnC{\equiv}CC_6H_5]_2\\&\quad + 2,4\text{-}C_7H_{12}\ (Ln = Gd,\ Er)\end{aligned} \quad (4)$$

During the reaction period, the color of the solution gradually changed from yellow to red, and red crystals of the phenylalkynide compound were obtained after suitable workup.

The IR spectra of Gd and Er compounds show strong absorption at 2037cm^{-1}, which is assigned to the absorption of $-C{\equiv}C-$ bond [15]. The mass spectra of the complexes did not give a parent molecular ion (2M), and gave only some fragments, such as [2M −

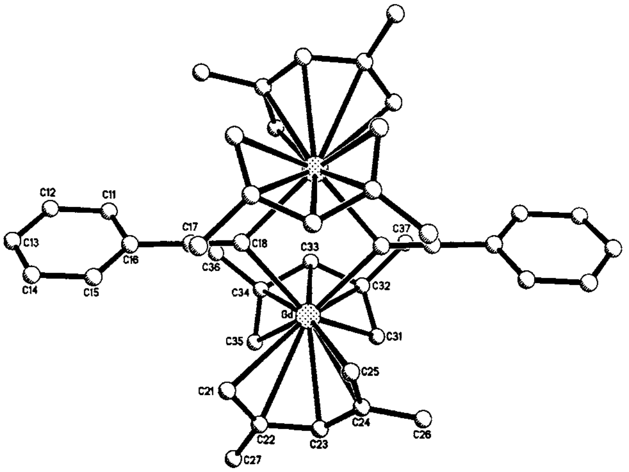


Fig. 1. The structure of $[(2,4\text{-}C_7H_{11})_2GdC{\equiv}CC_6H_5]_2$.

$C_7H_{11}]^+$, $[2M-C_8H_5]^+$, which denote the dimeric structure for Gd.

3.2. Molecular structures of the complexes

The structure of $[(2,4\text{-}C_7H_{11})_2LnC{\equiv}CC_6H_5]_2$ (Ln = Gd, Er) (Figs. 1 and 2) was established by single-crystal X-ray analysis. The complex is an alkynyl-bridged dimer, both the 2,4-dimethylpentadienyl ligands are nearly planar with maximum deviation from their least squares planes of 0.1371 (0.1379) for C(27) and 0.1026 (0.0902) for C(36), which form a dihedral angle of 123.3° (123.7°) between the two planes.

The $2,4\text{-}C_7H_{11}$ ligand adopts the planar U conformation. The C–C bond distances of Gd compound fall in two sets, viz. the external C–C bond distances, C(21)–C(22), C(24)–C(25), C(31)–C(32), C(34)–C(35), average 1.351(14) Å (1.341(15) Å); and the internal C–C bond distances, C(22)–C(23), C(23)–C(24), C(32)–C(33), C(33)–C(34), average 1.430(12) Å (1.425(13) Å). The difference of 0.079 Å (0.084 Å) between internal and external C–C bond distances is larger than that in $(2,4\text{-}C_7H_{11})_3Gd$ (0.046 Å) [2] or $(2,4\text{-}C_7H_{11})_3Er$ [3] (0.001Å).

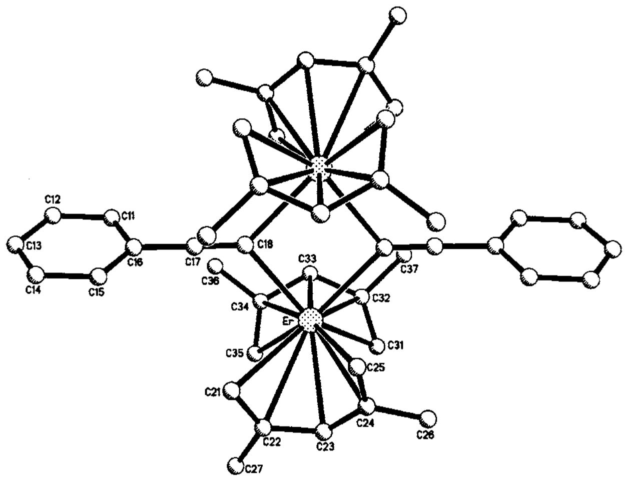


Fig. 2. The structure of $[(2,4\text{-}C_7H_{11})_2ErC{\equiv}CC_6H_5]_2$.

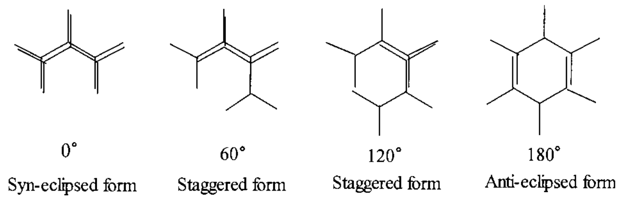


Fig. 3. Conformations of the two 2,4-dimethylpentadienyl ligands.

It is interesting to note that the $Gd\text{–}C(2,4\text{-}C_7H_{11})$ distances fall in two different orders, Gd–C(21,25) (2.866(10), 2.802(10) Å) > Gd–C(22,24) (2.814(8), 2.723(9) Å) > Gd–C(23) (2.673(8) Å) and Gd–C(32, 34) (2.788(9), 2.867(8) Å) > Gd–C(31, 35) (2.773(10), 2.730(9) Å) > Gd–C(33) (2.687(8) Å). It might be that the $C{\equiv}CC_6H_5$ ligand is situated against an open edge (C21–27) of a $2,4\text{-}C_7H_{11}$ ligand, which leads to repulsion between $C{\equiv}CC_6H_5$ and C_{31} as well as C_{35}, since it is situated against the back end (C31–37) of the other $2,4\text{-}C_7H_{11}$ ligand. The $Er\text{–}C(2,4\text{-}C_7H_{11})$ distances are essentially consistent with the above mentioned law.

The Gd–C(18) [Er–C(18)] and Gd–C(18a) [Er–C(18a)] distances are 2.512(8) (2.512(9)) and 2.566(8) (2.557(9) Å), respectively, which indicates that the electron deficient alkynyl bridge is asymmetrical, and the reason is the same as mentioned above. In the ring-opened cyclopentadienyl–lanthanide complex, the relationship between two ligand conformations is intriguing. The angle between Ln–C(23)–1/2[C(21) + C(25)] and Ln–C(33)–1/2 [C(31) + C(35)] planes is defined as the conformation angle when two ligands are *syn*-eclipsed the conformation angle is equal to 0°; when they are *anti*-eclipsed the conformation angle is 180°. The schemes are shown in Fig. 3.

For the transition metal sandwich compounds the conformation angles of $(2,4\text{-}C_7H_{11})_2M$ (M = V, Cr, Fe) are from 59.7 to 89.8° [10,19].

The complexes of Gd and Er have the conformation angles of 160.9 (Gd) and 157.9°(Er), which are greater than the transition metal sandwich compounds. Owing to steric hindrance, the two 2,4-dimethylpentadienyls adopt *anti*-eclipsed form (Fig. 3). The Gd–C≡C [Er–C≡C] and Gd_a–C≡C [Er_a–C≡C] angles are 125.6 (126.0°) and 135.6 (135.0°), respectively. The observed 9–10° difference is similar to that of $\{[(CH_3)N](CH_3)\text{-}Be(C{\equiv}CCH_3)\}$[20] (11°), but smaller than that of $\{[t\text{-}C_4H_9C_5H_4)_2SmC{\equiv}CC_6H_5]_2$ (58.2°) [14] and $[(C_5H_5)_2Er\text{-}C{\equiv}CC(CH_3)_3]_2$ (34°) [11].

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