

*Journal of Organometallic Chemistry*, 399 (1990) 247–254  
 Elsevier Sequoia S.A., Lausanne  
 JOM 21260

## Synthesis and X-ray crystal structure of a monopentamethylcyclopentadienyl derivative of gadolinium $\{ \text{Na}(\mu_2\text{-THF})[(\text{C}_5\text{Me}_5)\text{Gd}(\text{THF})]_2(\mu_2\text{-Cl})_3(\mu_3\text{-Cl})_2 \}_2 \cdot 6 \text{ THF}$

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(Received April 5th, 1990)

### Abstract

The reaction of  $\text{GdCl}_3$  with 1 equiv of  $\text{NaC}_5\text{Me}_5$  generates a neutral complex  $\text{C}_5\text{Me}_5\text{GdCl}_2(\text{THF})_3$  and a novel complex  $\{ \text{Na}(\mu_2\text{-THF})[(\text{C}_5\text{Me}_5)\text{Gd}(\text{THF})]_2(\mu_2\text{-Cl})_3(\mu_3\text{-Cl})_2 \}_2 \cdot 6\text{THF}$  which recrystallizes from THF in triclinic, the space group  $P\bar{1}$  with unit cell dimensions of  $a$  12.183(4),  $b$  13.638(6),  $c$  17.883(7) Å,  $\alpha$  110.38(3),  $\beta$  94.04(3),  $\gamma$  99.44(3)°,  $V$  2721.20 Å<sup>3</sup> and  $D_{\text{calc}}$  1.43 g cm<sup>-3</sup> for  $Z=1$ . Least-squares refinement of 2170 observed reflections led to a final  $R$  value of 0.047. The title complex consists of two  $\text{Na}(\mu_2\text{-THF})[(\text{C}_5\text{Me}_5)\text{Gd}(\text{THF})]_2(\mu_2\text{-Cl})_3(\mu_3\text{-Cl})_2$  units bridged together via two  $\mu_2\text{-THF}$  to Na coordination. Each Gd ion is surrounded by one  $\text{C}_5\text{Me}_5$  ligand, two  $\mu_3\text{-Cl}$ , two  $\mu_2\text{-Cl}$  and one THF in a distorted octahedral arrangement with average Gd–C(ring) 2.686(33), Gd– $\mu_2\text{-Cl}$  2.724(7), Gd– $\mu_3\text{-Cl}$  2.832(8) and Gd–O 2.407(11) Å. The sodium ion coordinates to two bridging THF, two  $\mu_2\text{-Cl}$  and two  $\mu_3\text{-Cl}$  to form a distorted octahedron with average Na– $\mu_2\text{-O}$ , Na– $\mu_2\text{-Cl}$  and Na– $\mu_3\text{-Cl}$  of 2.411(21), 2.807(15) and 2.845(12) Å, respectively.

### Introduction

Since Wayda and Evans [1], and Tilley and Anderson [2] independently synthesized bispentamethylcyclopentadienyl lanthanide chloride, the synthesis and molecular structure of bispentamethylcyclopentadienyl lanthanide derivatives have been widely studied [3–8].

In contrast, few reports on the synthesis and molecular structure of monopentamethylcyclopentadienyl lanthanide derivatives  $(\text{C}_5\text{Me}_5)\text{LnCl}_2$  have appeared. The first derivatives of this type  $(\text{C}_5\text{Me}_5)\text{NdCl}_3\text{Na}(\text{Et}_2\text{O})_2$  [2] and  $(\text{C}_5\text{Me}_5)\text{YbCl}_3\text{Li}(\text{THF})$  [5] were synthesized in 1981. Recently, the synthesis of following complexes  $(\text{C}_5\text{Me}_5)\text{LnCl}_3\text{Li}(\text{THF})_2$  ( $\text{Ln} = \text{Y}, \text{La}, \text{Ce}$ ) [9,10],  $(\text{C}_5\text{Me}_5)\text{LnI}_2(\text{THF})_3$  ( $\text{Ln} = \text{La}, \text{Ce}$ ) [10],  $(\text{C}_5\text{Me}_5)\text{LnCl}_3\text{Na}(\text{Et}_2\text{O})_2$  ( $\text{Ln} = \text{Pr}, \text{Lu}$ ) and  $[\text{K}(\text{DME})_3]\{ \text{K}[(\text{C}_5\text{Me}_5\text{-Yb})_3\text{Cl}_8\text{K}(\text{DME})_2]_2 \}$  [11] have also been reported. However, only two of them,  $(\text{C}_5\text{Me}_5)\text{CeI}_2(\text{THF})_3$  and  $[\text{K}(\text{DME})_3]\{ \text{K}[(\text{C}_5\text{Me}_5\text{-Yb})_3\text{Cl}_8\text{K}(\text{DME})_2]_2 \}$  have been characterized by X-ray crystallography.

We have studied the reaction of  $\text{GdCl}_3$  and  $\text{NaC}_5\text{Me}_5$  in a 1:1 mole ratio and have successfully isolated and determined the structure of a novel monopentamethylcyclopentadienylgadolinium derivative  $\{\text{Na}(\mu_2\text{-THF})[(\text{C}_5\text{Me}_5)\text{Gd}(\text{THF})]_2(\mu_2\text{-Cl})_3(\mu_3\text{-Cl})_2\}_2 \cdot 6\text{THF}$ . We report here the details of the molecular structure.

## Experimental

All manipulations were performed by use of Schlenk techniques under dry, oxygen-free argon. THF was distilled from sodium benzophenone ketyl. Anhydrous  $\text{GdCl}_3$  was prepared from the hydrate by the method of Taylor and Carter [12].  $\text{NaC}_5\text{Me}_5$  was prepared from the reaction of  $\text{C}_5\text{Me}_5\text{H}$  and  $\text{NaH}$  in THF. Metal analyses were carried out by complexometric titration, chlorine by titration with 0.014 *N*  $\text{AgNO}_3$  solution and Na using a Shimadzu 646 atomic absorption spectrometer. Infrared spectra were recorded as KBr pellets with a Digilab FTS-20E spectrometer.

### $\{\text{Na}(\text{THF})[(\text{C}_5\text{Me}_5)\text{Gd}(\text{THF})]_2\text{Cl}_5\}_2 \cdot 6\text{THF}$

To a suspension of  $\text{GdCl}_3$  (4.80 g, 18.2 mmol) in THF (50 ml) was added with stirring a solution of  $\text{NaC}_5\text{Me}_5$  (3.64 g, 18.3 mmol) in THF (50 ml). The mixture, which turned a turbid yellow, was stirred for 72 h at room temperature. After centrifugation, the filtrate was concentrated to 20 ml and cooled to  $-10^\circ\text{C}$ . White crystals of  $\{\text{Na}(\text{THF})[(\text{C}_5\text{Me}_5)\text{Gd}(\text{THF})]_2\text{Cl}_5\}_2 \cdot 6\text{THF}$  were obtained in 22.9% yield (2.55 g). Anal. Found: Gd, 25.80; Cl, 14.29; Na, 1.90.  $\text{Gd}_4\text{Na}_2\text{Cl}_{10}\text{C}_{88}\text{H}_{156}\text{O}_{12}$  calc.: Gd, 25.83; Cl, 14.58; Na, 1.89%. IR(KBr): 2870(s), 1375(m), 1245(w), 1180(m), 1060(s), 1040(s), 1020(s), 910(s), 865(m)  $\text{cm}^{-1}$ .

### $(\text{C}_5\text{Me}_5)\text{GdCl}_2(\text{THF})_3$

The mother liquor from the above reaction was concentrated to 10 ml and cooled to  $-30^\circ\text{C}$ . White crystals (1.0 g, 10%) were obtained. Anal. Found: Gd, 27.13; Cl, 12.62.  $\text{GdC}_{22}\text{H}_{39}\text{Cl}_2\text{O}_3$  calc.: Gd, 27.20; Cl, 12.50%. IR(KBr): 2870(s), 1374(m), 1245(w), 1179(m), 1062(s), 1040(s), 1020(s), 910(s), 860(m)  $\text{cm}^{-1}$ .

### *X-Ray crystallography of $\{\text{Na}(\text{THF})[(\text{C}_5\text{Me}_5)\text{Gd}(\text{THF})]_2\text{Cl}_5\}_2 \cdot 6\text{THF}$*

A single crystal of  $0.28 \times 0.37 \times 0.36$  mm was sealed under argon in a thin-walled glass capillary. The unit cell parameters were determined from the setting angles of a number of reflections. Data were collected on a Nicolet R3M/E diffractometer using graphite monochromated  $\text{Mo-K}_\alpha$  radiation. A total of 5151 intensities within the range of  $3^\circ < 2\theta < 40^\circ$  were measured by use of the  $\omega$ -scan technique. A summary of data collection parameters is given in Table 1.

The intensities were corrected for Lorentz and polarization effects, but not for absorption. The gadolinium atoms were located by Patterson technique and the remaining non-hydrogen atoms were located by successive Fourier syntheses. All positional parameters and temperature factors for non-hydrogen atoms were refined anisotropically. The final *R* is 0.047,  $R_w = 0.047$ . Final positional parameters are given in Table 2. All calculations were carried out with the SHELXTL computer program system.

Table 1

## Crystal data and experimental parameters

Compound	Gd <sub>4</sub> Na <sub>2</sub> Cl <sub>10</sub> C <sub>88</sub> H <sub>156</sub> O <sub>12</sub>
Molecular weight	2345.77
Crystal size, mm	0.28 × 0.37 × 0.36
Cell constants	
<i>a</i> , Å	12.183(4)
<i>b</i> , Å	13.638(6)
<i>c</i> , Å	17.883(7)
$\alpha$ , deg	110.38(3)
$\beta$ , deg	94.04(3)
$\gamma$ , deg	99.44(3)
<i>V</i> , Å <sup>3</sup>	2721.20
<i>D</i> , g/cm <sup>3</sup>	1.43
<i>Z</i>	1
scan technique	$\omega$
2 $\theta$ range, deg	3–40
No. of reflections measured	5151
Space group	<i>P</i> $\bar{1}$
No. of reflections for $I < 3\sigma(I)$	2170
<i>F</i> (000)	402
$R = \sum   F_o  -  F_c   / \sum  F_o $	0.047
$R_w = (\sum w(F_o - F_c)^2 / \sum wF_o^2)^{1/2}$	0.042

*Supplementary material available.* Tables listing the thermal parameters, bond distances and angles, best planes (21 pages) are available from the authors.

## Results and discussion

### Synthesis

CeI<sub>3</sub> and YbCl<sub>3</sub> have been reported to react with 1 equivalent of KC<sub>5</sub>Me<sub>5</sub> to form (C<sub>5</sub>Me<sub>5</sub>)CeI<sub>2</sub>(THF)<sub>3</sub> [10] and [K(DME)<sub>3</sub>]{K[(C<sub>5</sub>Me<sub>5</sub>Yb)<sub>3</sub>Cl<sub>8</sub>K(DME)<sub>2</sub>]<sub>2</sub>} [11], respectively, which have been crystallographically characterized. LnCl<sub>3</sub> (Ln = Nd, Pr, Yb, Lu) has also been reported to react with 1 equivalent of NaC<sub>5</sub>Me<sub>5</sub> or LiC<sub>5</sub>Me<sub>5</sub> in THF or Et<sub>2</sub>O to produce (C<sub>5</sub>Me<sub>5</sub>)LnCl<sub>3</sub>M(THF)<sub>2</sub> or (C<sub>5</sub>Me<sub>5</sub>)LnCl<sub>3</sub>M(Et<sub>2</sub>O)<sub>2</sub> (M = Na or Li) [2,5,9–11]. However, the molecular structure has not yet been determined. In our study on the reaction of GdCl<sub>3</sub> and NaC<sub>5</sub>Me<sub>5</sub> in 1:1 mole ratio in THF, the first crop of crystals is a THF-bridged dimer {Na(THF)[(C<sub>5</sub>Me<sub>5</sub>)Gd(THF)<sub>2</sub>Cl<sub>5</sub>]<sub>2</sub> · 6THF. The second crop of crystals from the mother liquor is the neutral complex (C<sub>5</sub>Me<sub>5</sub>)GdCl<sub>2</sub> · 3THF identified from its elemental analysis and infrared spectra. The complex {Na(THF)[(C<sub>5</sub>Me<sub>5</sub>)Gd(THF)<sub>2</sub>Cl<sub>5</sub>]<sub>2</sub> · 6THF, decomposes at temperatures above 23°C losing THF.

### Crystal structure

The complex {Na(THF)[(C<sub>5</sub>Me<sub>5</sub>)Gd(THF)<sub>2</sub>Cl<sub>5</sub>]<sub>2</sub> · 6THF consists of two monomers {Na( $\mu_2$ -THF)[(C<sub>5</sub>Me<sub>5</sub>)Gd(THF)<sub>2</sub>( $\mu_2$ -Cl)<sub>3</sub>( $\mu_3$ -Cl)<sub>2</sub>] which are connected by two THF bridges. To our knowledge, this is the first structure of an organo-lanthanocene dimer held together by bridging THF molecules. Six THF molecules are present in the crystal lattice as solvate molecules. The structure is depicted in

Table 2

Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\times 10^3$ ) (e.s.d.'s refer to last significant digit)

Atom	x	y	z	U
Gd1	2291(1)	2114(1)	1981(1)	61(1)
Gd2	4792(1)	1484(1)	3202(1)	62(1)
Na	4436(11)	588(8)	904(5)	81(6)
C11	4583(7)	2614(5)	2093(3)	67(4)
C12	3023(7)	2571(5)	3590(3)	73(4)
C13	3040(7)	212(5)	2052(4)	72(4)
C14	2373(7)	986(5)	445(3)	84(4)
C15	6097(8)	647(6)	2116(4)	88(4)
O1	622(18)	938(13)	2018(9)	87(6)
C11	383(27)	834(20)	2781(15)	87(9)
C12	-698(34)	-75(24)	2496(18)	127(12)
C13	-955(31)	-575(22)	1586(16)	104(11)
C14	-45(30)	-27(22)	1260(16)	96(10)
C15	2252(30)	3784(21)	1518(16)	87(10)
C16	2450(29)	4237(20)	2390(15)	83(9)
C17	1480(31)	3904(23)	2662(17)	95(10)
C18	681(33)	3218(23)	1975(19)	100(11)
C19	1115(33)	3198(23)	1309(18)	100(11)
C20	3152(32)	3964(23)	935(17)	107(11)
C21	3487(35)	4985(25)	2893(18)	132(12)
C22	1379(31)	4363(22)	3601(17)	104(11)
C23	-590(37)	2880(27)	2054(20)	144(14)
C24	565(31)	2730(23)	468(17)	108(11)
C31	5828(29)	455(21)	3962(15)	79(9)
C32	4733(23)	-132(20)	3675(14)	68(8)
C33	3931(27)	462(21)	4127(14)	78(9)
C34	4603(27)	1386(21)	4665(14)	71(8)
C35	5793(27)	1405(21)	4593(15)	73(9)
C36	6926(30)	71(22)	3749(16)	94(10)
C37	4482(30)	-1315(22)	3004(16)	105(10)
C38	2640(32)	24(24)	3974(37)	103(11)
C39	4109(28)	2239(21)	5320(16)	108(11)
C40	6724(25)	2250(19)	5142(14)	91(9)
O3	6146(15)	3152(11)	3772(8)	81(8)
C41	5889(31)	4249(22)	4269(17)	131(12)
C42	6744(35)	5043(25)	4141(19)	145(14)
C43	7839(33)	4438(25)	3983(18)	142(14)
C44	7263(28)	3296(20)	3455(15)	104(11)
O5	5628(19)	1174(13)	49(10)	77(6)
C51	5119(29)	1809(24)	-295(16)	101(10)
C52	6101(40)	2612(33)	-288(25)	195(18)
C53	7124(46)	2637(37)	274(29)	245(24)
C54	6777(30)	1687(26)	433(17)	104(12)
O6	6638(47)	6292(43)	2390(27)	347(24)
C61	7282(56)	7180(42)	2230(36)	256(29)
C62	6813(59)	6751(47)	1396(34)	264(27)
C63	6009(51)	5673(50)	1169(32)	265(28)
C64	6858(54)	5412(44)	1662(40)	268(30)
O7	10094(53)	6155(46)	1957(32)	435(41)
C71	10962(66)	7148(47)	2240(34)	305(29)
C72	11902(65)	6812(66)	1882(56)	349(41)

Table 2 (continued)

Atom	x	y	z	U
C73	11172(69)	6507(55)	1076(39)	300(35)
C74	10050(50)	5791(43)	1096(30)	287(24)
O8	9483(43)	8071(34)	4210(25)	382(33)
C81	9149(49)	6989(38)	4173(26)	291(23)
C82	10100(48)	6886(49)	4870(36)	318(35)
C83	10160(48)	7908(42)	5345(32)	224(26)
C84	10080(38)	8685(30)	5033(23)	271(20)

Fig. 1. For clarity the THF molecules in the Fig. 1 are represented by the O atoms only. Bond lengths and bond angles are listed in Tables 3 and 4.

Two gadolinium atoms and one sodium atom in each monomer form an approximately equilateral triangle with angles Gd1–Gd2–Na 58.3(2), Gd1–Na–Gd2 63.1(2), Gd2–Gd1–Na 58.5(2)°. Three chlorine atoms bridge the edges of the triangle with angles Gd1–Cl2–Gd2 91.8(2), Gd1–Cl4–Na 89.0(3), Gd2–Cl5–Na 88.3(4)°, while two additional chlorine atoms cap the Gd1Gd2Na fragment on each side in  $\eta^3$ -fashion. Fig. 2 shows the coordination geometries around the Gd–Na cluster. Each gadolinium ion is coordinated to one  $\eta^5$ -C<sub>5</sub>Me<sub>5</sub> group, one THF oxygen atom, two  $\mu_2$ -Cl and two  $\mu_3$ -Cl giving the metal a formal coordination number of 8. The geometry around the gadolinium ion is a distorted octahedron.

The average angle Cent–Gd–O (Cent = centroid of C<sub>5</sub>Me<sub>5</sub>) 103.3(4)° is in the range normally observed for bis(pentamethylcyclopentadienyllanthanide [6] and monopentamethylcyclopentadienyllanthanide [10]. The ring carbon atoms all lie within 0.0122 Å of the calculated C31–35 mean plane. The Gd–C(ring) distances

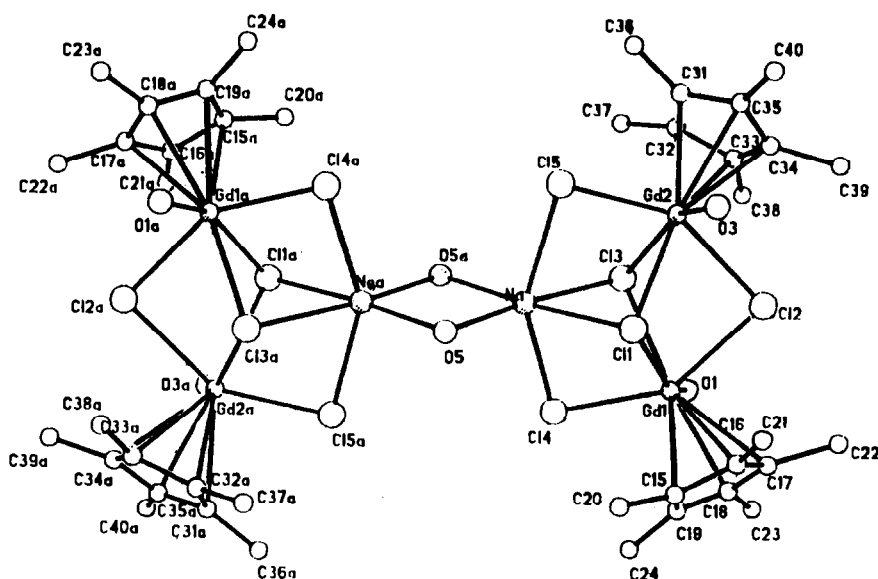


Fig. 1. Molecular structure of  $\{Na(\mu_2\text{-THF})(C_5Me_5)Gd(THF)\}_2(\mu_2\text{-Cl})_3(\mu_3\text{-Cl})_2\}_2 \cdot 6THF$ .

Table 3

Selected bond distances (Å) (e.s.d.'s refer to last significant digit)

Gd1–Gd2	4.002(2)	Gd1–Na	3.818(12)
Gd1–Cl1	2.740(8)	Gd1–Cl2	2.769(6)
Gd1–Cl3	2.928(8)	Gd1–Cl4	2.660(6)
Gd1–O1	2.394(20)	Gd1–Cl5	2.686(33)
Gd1–Cl6	2.697(28)	Gd1–Cl7	2.703(33)
Gd1–Cl8	2.662(40)	Gd1–Cl9	2.714(39)
Gd2–Na	3.826(9)	Gd2–Cl1	2.925(8)
Gd2–Cl2	2.805(9)	Gd2–Cl3	2.734(6)
Gd2–Cl5	2.662(8)	Gd2–C31	2.680(34)
Gd2–C32	2.614(30)	Gd2–C33	2.674(31)
Gd2–C34	2.691(28)	Gd2–C35	2.738(29)
Gd2–O3	2.420(1)	Na–Cl1	2.807(10)
Na–Cl3	2.883(14)	Na–Cl4	2.785(16)
Na–Cl5	2.829(14)	Na–O5	2.422(24)
Na–Naa	3.558(20)	Na–O5a	2.399(18)
C13–C14	1.500(49)	C15–C16	1.449(36)
C16–C17	1.378(50)	C15–C19	1.433(49)
C17–C18	1.438(40)	C18–C19	1.329(51)
C31–C32	1.396(42)	C31–C35	1.400(34)
C32–C33	1.477(44)	C33–C34	1.375(32)
C34–C35	1.462(47)	O5–Naa	2.399(18)
Gd1–C(ring1)	2.692(35)	Gd2–C(ring2)	2.679(30)
Gd1–Cent1 <sup>a</sup>	2.441(2)	Gd2–Cent2	2.387(2)

<sup>a</sup> Cent = centroid of pentamethylcyclopentadienyl.

range from 2.614(30) to 2.738(29) and average 2.686(33) Å, which is comparable to 2.72 Å in  $(C_5Me_5)_2SmCl \cdot THF$ , 2.78 Å in  $(C_5Me_5)_2PrCl_2Na(DME)_2$  and 2.797 Å in  $(C_5Me_5)CeI_2(THF)_3$ , if the atomic radius of Gd, Sm, Pr, Ce are considered.

The Gd– $\mu_2$ –Cl distances can be divided into two groups. (i) Gd– $\mu_2$ –Cl bonds which are connected to the other Gd atom are Gd1–Cl2 2.769(6) Å and Gd2–Cl2 2.805(9) Å, while Gd– $\mu_2$ –Cl bonds connect to the Na atom, Gd1–Cl4 2.660(6) Å, Gd2–Cl5 2.662(8) Å, are shorter. Similar behavior has been observed for the

Table 4

Selected bond angles (°) (e.s.d.'s refer to last significant digit)

Gd1–Gd2–Na	58.3(2)	Gd2–Gd1–Na	58.5(2)
Gd1–Na–Gd2	63.1(2)	Cl4–Na–Cl5	150.5(4)
O5–Na–O5a	84.9(7)	Na–O5–Naa	95.1(7)
Na–Cl5–Gd2	88.3(4)	Na–Cl4–Gd1	89.0(3)
Gd1–Cl2–Gd2	91.8(2)	Gd1–Cl1–Gd2	89.8(2)
Gd1–Cl1–Na	87.0(3)	Gd2–Cl1–Na	83.7(3)
Gd1–Cl3–Na	82.1(3)	Gd1–Cl3–Gd2	89.9(2)
Gd2–Cl3–Na	85.8(3)	Cent1–Gd1–O1	104.0(7)
Cent1–Gd1–Cl1	106.6(2)	Cent1–Gd1–Cl2	106.7(2)
Cent1–Gd1–Cl3	178.0(2)	Cent1–Gd1–Cl4	103.0(3)
Cent2–Gd2–O3	102.6(1)	Cent2–Gd2–Cl1	174.8(2)
Cent2–Gd2–Cl2	104.1(2)	Cent2–Gd2–Cl3	108.0(2)
Cent2–Gd2–Cl5	106.1(3)		

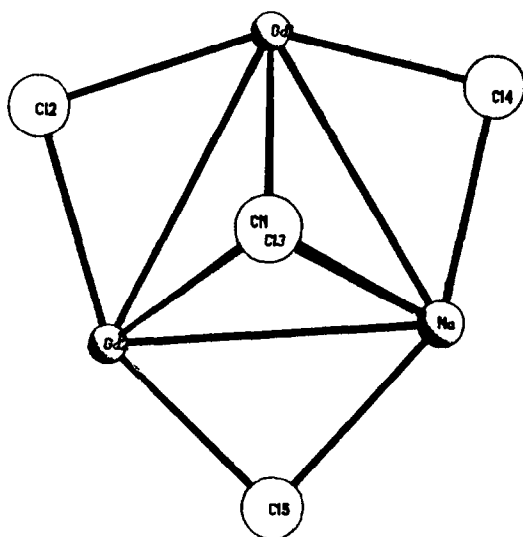


Fig. 2. The coordination geometries around the Gd-Na cluster.

Ln- $\mu$ -Cl distance in the complexes with a Ln( $\mu$ -Cl)<sub>2</sub>Ln unit or a Ln( $\mu$ -Cl)<sub>2</sub>M unit [11].

The geometry around sodium can be regarded as a distorted octahedron formed by two  $\mu_2$ -Cl, two  $\mu_3$ -Cl and two  $\mu_2$ -O of two THF molecules. The two sodium atoms are connected by two O bridges with angles Na-O5-Na 95.1(7), O5-Na-O5a 84.3(10)°. The Na-Cl distances fall into two ranges. The distances of Na to doubly-bridging Cl, Na-Cl4, 2.785(16) Å, and Na-Cl5, 2.829(14) Å, are in the range expected, such as that in (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>PrCl<sub>2</sub>Na(DME)<sub>2</sub> [4]. The distances of Na to triply-bridging Cl, Na-Cl1, 2.807(11) Å, Na-Cl3, 2.883(14) Å, are somewhat longer. This is usual since increased bridging tends to give longer bond length in the complexes of this type [13,14]. The average Na- $\mu$ -O distance is 2.411(21) Å which is shorter than 2.387(12) Å found in [Na(THF)<sub>6</sub>][(C<sub>5</sub>H<sub>5</sub>)<sub>3</sub>Nd( $\mu$ -H)Nd(C<sub>5</sub>H<sub>5</sub>)<sub>3</sub>] · 2THF [15]. The lengthening of a bond to a bridging oxygen atom is reasonable.

### Acknowledgement

The authors express their thanks to the Chinese National Foundation of Natural Science for financial support.

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