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Journal of Alloys and Compounds 408–412 (2006) 1030–1036

Journal of ALLOYS AND COMPOUNDS

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Lanthanide complexes of some high energetic compounds (III), crystal structures and thermal properties of 2,6-dinitrophenol (2,6-DNP) complexes

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Received 30 July 2004; received in revised form 20 November 2004; accepted 14 December 2004
Available online 15 July 2005

Abstract

The Ln(III) complexes with 2,6-dinitrophenol (2,6-DNP) ligand, $[La_2(2,6\text{-DNP})_6(H_2O)_4]\cdot 4H_2O$, **1**, and $[Tb(2,6\text{-DNP})_3(H_2O)_3]$, **2**, have been synthesized and their crystal structures have been analyzed by X-ray diffraction methods. Complex **1** and **2**, both crystallize in the triclinic space group $P\bar{1}$. In complex **1**, 2,6-DNP acts as the bidentate. The structural feature is the presence of the bridged ligands and the terminal ligands in the bimetallic structure. In addition, there are four uncoordinated water molecules in the crystal lattice. The 10 coordinated La(III) ion forms a bicapped square antiprism. In complex **2**, three 2,6-DNP ligands coordinate directly to the metal ion in the bidentate fashion. The nine coordinated Tb(III) ion forms slightly distorted tricapped trigonal prism. There are no water molecules in the crystal lattice. Based on the results of TG-DTG and DSC thermal analysis, it was analyzed that the lanthanide 2,6-DNP complexes are thermally decomposed in three distinctive stages, the dehydration, the 2,6-DNP decomposition, and the formation of the metal oxide.

Keywords: Lanthanide complex; 2,6-Dinitrophenol; Thermal analysis; Crystal structure

1. Introduction

In the previous paper, we have reported the crystal structures and thermal properties of samarium and holmium picrates, among the lighter and heavier lanthanide series, respectively [1]. Unlikely with that reported [2], it was found that the samarium picrate complex appears to be such a dodecahydrated complex like other members of lighter lanthanide series [3].

We have found that the coordination behavior of 2,6-dinitrophenol (2,6-DNP) is quite different with that of the picric acid by the studies of yttrium and neodymium complexes of 2,6-DNP [4,5]. It was known that the electronic effect of the nitro substituent at para-position influences to the coordination behavior of the nitrophenols as the ligands. As the extension of the studies to confirm the coordination behavior of the nitrophenols, we have investigated the X-ray single crystal structures and thermal properties of lanthanide complexes of 2,6-DNP, $[La_2(2,6\text{-DNP})_6(H_2O)_4]\cdot 4H_2O$, **1**, and $[Tb(2,6\text{-DNP})_3(H_2O)_3]$, **2**. And thermal properties of yttrium and some other lanthanide complexes have also studied.

2. Experimental

2.1. Preparation and analysis of the complexes

The complexes were prepared with the process similar to the preparation of the picrate complexes [1]. The metal

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doi:10.1016/j.jallcom.2004.12.184

solutions were prepared by dissolving respective lanthanide chlorides ($LnCl_3 \cdot nH_2O$, Aldrich; ca. 0.84 mmol) in distilled water (20 mL) at 60 °C with stirring under the water bath. The pH of the solution was adjusted to ca. 2 with 7 M HNO_3 solution. The ligand solution was prepared by dissolving 2,6-dinitrophenol ($C_6H_4N_2O_5$, moist solid with 20% water, Merck; 0.3 g, 1.3 mmol) in distilled water (30 mL) at 60 °C with stirring under the water bath. The pH of the solution was adjusted to about 6 with 3 M LiOH solution. The metal solution was added dropwise slowly to the ligand solution. The reaction mixture was stirred for 3 h at 60 °C adjusting its pH to ca. 4 and then filtrated through a 0.45-μm plastic membrane in order to remove the small amount of the precipitate formed. The final solution was cooled and kept in the refrigerator. Light yellow crystals of the 2,6-DNP complexes of respective metal ions were obtained at room temperature over a period of a few weeks. The complexes were recrystallized from distilled water. Upon cooling of hot aqueous solutions of the complexes in the refrigerator, the crystals of complexes suitable for single crystal X-ray diffraction were obtained.

The contents of carbon, hydrogen, and nitrogen were determined by a CE EA-1110 elemental analyzer. The complexes are relatively stable in ambient condition, so the results of the elemental analysis are well agreed with the calculated values. Anal. calcd. (%) for $C_{36}H_{34}N_{12}O_{38}La_2$: C, 28.44; H, 2.25; N, 11.05. Found: C, 28.13; H, 2.12; N, 10.95. Anal. calcd. (%) for $C_{18}H_{15}N_6O_{18}Nd$: C, 28.91; H, 2.01; N, 11.24. Found: C, 28.82; H, 2.09; N, 11.53. Anal. calcd. (%) for $C_{18}H_{15}N_6O_{18}Tb$: C, 28.35; H, 1.97; N, 11.03. Found: C, 28.86; H, 1.98; N, 10.92. Anal. calcd. (%) $C_{18}H_{15}N_6O_{18}Y$: C, 31.21; H, 2.17; N, 12.14. Found: C, 30.37; H, 2.18; N, 11.06.

2.2. *X-ray crystallography*

The data for X-ray structure determination were collected on a Enraf-Nonius CAD-4 X-ray diffractometer equipped with graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at 293 K. The unit cell dimensions were determined on the basis of 25 reflections in the range of $11.26° < \theta < 13.07°$ and $11.93° < \theta < 13.70°$ for Ln(III)- and Tb(III)-2,6-DNP, respectively. The data were collected by the $\omega - 2\theta$ technique. Empirical absorption correction was applied to the intensity data. The standard direct method was used to position the heavy atoms. The remaining non-hydrogen atoms were located from the subsequent difference Fourier synthesis. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were calculated in ideal positions and were riding on their respective carbon atoms ($B_{iso} = 1.2B_{eq}$). The structure was refined in a full matrix least-squares calculation on F^2. Program used to solve structure and to refine structure: SHELXS97 and SHELXL97 [6]. Molecular graphics; ORTEP-3 [7]. Crystallographic and experimental data for the complexes **1** and **2** are presented in detail in Table 1.

2.3. *Thermal analysis*

The thermal decomposition of the complex was investigated on a Mettler-Toledo TGA 50 apparatus and Mettler-Toledo DSC model 821^e apparatus. The experimental procedure for the thermal analysis is similar to that described previously [1].

3. Results and discussion

3.1. *Crystal structure*

The lanthanum 2,6-DNP complex appeared to be an unique octa-hydrated dinuclear complex whose stoichiometric chemical formula is $[La_2(2,6\text{-DNP})_6(H_2O)_4] \cdot 4H_2O$, **1**. It is comprising a symmetric neutral dimer $[La_2(2,6\text{-DNP})_6(H_2O)_4]$ and four water molecules in the outer coordination sphere. The formation of dimeric complex is unusual since yttrium [4] and other members of lanthanide, such as neodymium [5] and terbium, form the monomeric neutral complexes with 2,6-DNP.

Table 1
Crystal data and structure refinement for complexes **1** and **2**

Compound	**1**	**2**
Empirical formula	$C_{36}H_{34}N_{12}O_{38}La_2$	$C_{18}H_{15}N_6O_{18}Tb$
Formula weight	1520.57	762.28
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions		
a (Å)	9.162(1)	8.5916(8)
b (Å)	12.252(6)	11.734(2)
c (Å)	12.570(1)	13.809(2)
α (°)	107.60(2)	73.79(2)
β (°)	100.74(1)	74.66(1)
γ (°)	96.98(2)	69.44(2)
Volume ($Å^3$)	1297.5(7)	1230.0(3)
Z	1	2
Calculated density (g/cm^3)	1.946	2.058
Absorption coefficient (mm^{-1})	1.750	2.979
F(0 0 0)	752	748
θ range for data collection (°)	2.04–24.97	2.19–24.97
Index ranges	$-10 \le h \le 10$, $-14 \le k \le 13$, $0 \le l \le 14$	$-9 \le h \le 10$, $-13 \le k \le 13$, $0 \le l \le 16$
Reflections collected/unique	4739/4516 ($R_{int} = 0.0271$)	4502/4308 ($R_{int} = 0.0558$)
Data/restraints/parameters	4516/0/407	4308/0/379
Goodness-of-fit on F^2	1.057	1.028
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0283$, $wR_2 = 0.0741$	$R_1 = 0.0523$, $wR_2 = 0.1513$
R indices (all data)	$R_1 = 0.0305$, $wR_2 = 0.0755$	$R_1 = 0.0548$, $wR_2 = 0.1542$
Largest diff. peak and hole ($eÅ^{-3}$)	1.454 and −0.768	2.513 and −2.305

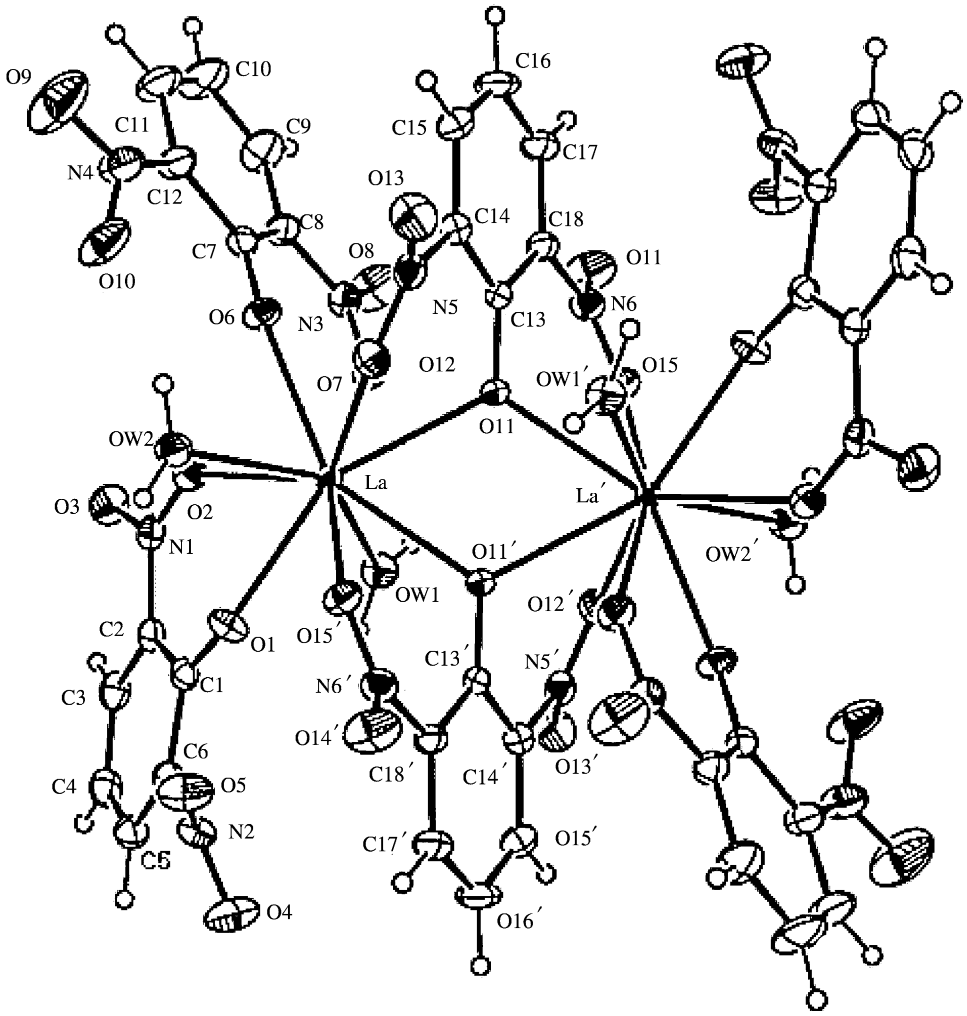

Fig. 1. The molecular structure of $[La_2(2,6\text{-}DNP)_6(H_2O)_4]\cdot 4H_2O$, **1**, showing the atom numbering scheme and 30% probability displacement ellipsoids.

The selected bond lengths and angles for the complex **1** are listed in Table 2. An ORTEP diagram of the molecular structure for the complex **1** is presented by the atom numbering scheme in Fig. 1.

Table 2
Selected bond lengths (Å) and angles (°) for complex **1**

La—O6	2.391(2)	O1—C1	1.275(4)
La—O1	2.405(2)	O2—N1	1.245(4)
La—O11′	2.521(2)	O6—C7	1.257(4)
La—O11	2.534(2)	O7—N3	1.245(4)
La—Ow2	2.561(3)	O8—N3	1.225(4)
La—Ow1	2.586(2)	O11—C13	1.302(4)
La—O2	2.636(3)	O11—La′	2.521(2)
La—O7	2.666(2)	O12—N5	1.235(4)
La—O12	2.764(2)	O15—La′	2.837(2)
La—O15′	2.837(2)		
O11′—La—O11	60.85(8)	N1—O2—La	137.0(2)
O11—La—O12	60.31(7)	C7—O6—La	148.1(2)
O1—La—O2	64.11(8)	N3—O7—La	141.5(2)
O6—La—O7	62.28(8)	C13—O11—La	119.0(2)
O11′—La—O15′	58.65(7)	La′—O11—La	119.15(8)
C1—O1—La	139.1(2)	N5—O12—La	129.8(2)

As shown in Fig. 1, the important structural feature of the complex **1** is the presence of the bridged ligands and the terminal ligands in the bimetallic structure. It is the dimeric structure in centrosymmetry excluding outersphere water molecules. There are four water molecules coordinated directly, two to each lanthanum metal ion. In addition, there are four water molecules (Ow3, Ow4, Ow3′, and Ow4′) in the outer sphere. The faces of the two ligands at the terminal site are twisted in the direction of 90° with respect to each other.

Each La(III) ion is coordinated to 10 O atoms. Two of these neighbors are the O atoms of water molecules (Ow1 and Ow2). The $La–O_{water}$ bond distances are within the range of 2.561(3)–2.586(2) Å and are longer than the sum of the covalent radii of the La and O atoms (2.53 Å). Each 2,6-DNP ligand forms a chelate ring with an La(III) ion through the O atoms of one nitro group and the phenol group. The $[La_2(2,6\text{-}DNP)_6(H_2O)_4]$ has eight six-membered chelate rings composed of two La(III) ions, four terminal ligands, and two bridged ligands. The terminal ligand behaves as a bidentate ligand involving in the chelate formation through

O atoms of a phenol group and a nitro group. The bridged ligand behaves as a terdentate ligand that an O atom of the phenol group bridges to two metal ions and each O atoms of two nitro groups bind to each other of two metal ions.

Considering the distances of the La–O bonds in detail, the La–O distances to the nitro groups in the range of 2.636(3)–2.837(2) Å are considerably longer than the La–O distances to the phenol groups in the range of 2.391(2)–2.534(2) Å, which means that La–O bonds to the nitro groups have relatively weak strength.

Some remarkable features have been observed for bridged ligands composed of La–O12, La–O15′, and La–O11. The distance between La and O12 atoms of nitro groups is 2.764(2) Å, whereas the distance between La and O15′ atoms of nitro groups is 2.837(2) Å. The difference in the distance would be come out from that the former O12 atom is placed on basal position while the later O15 atom on capped position in the complex polyhedron. The bond distances of La–O11 and La′–O11 are 2.534(2) and 2.521(2) Å, respectively. On the other hand, there is no metal–metal interaction since the distance of La–La′ atoms is 4.36 Å. The distance for O11–C13 of the phenol group in the bridged ligand is 1.302 Å, which is a little longer than the corresponding distance (1.275(4) Å for O1–C1) in the terminal ligand. The nitro groups of the bridged ligand are twisted in the center of C13.

The calculated dihedral angles between the benzene rings and the nitro groups are good evidence for an interaction between the O atoms on the nitro groups and the La atom. The dihedral angles are in the range 1.7(3)°–10.9(2)° for the coordinated nitro groups and 13.0(4)°–35.9(1)° for the uncoordinated nitro groups in the terminal ligands. However, the dihedral angles in the bridged ligands, of which both nitro groups are coordinated, are rather twisted in the range 26.5(2)°–31.1(2)°. Considering one of the terminal 2,6-DNP ligands of an La(III) ion in Fig. 1, the closest approach to La of the O atoms of the uncoordinated nitro groups is 4.74 Å, while the bond distance to La of the O atoms of the coordinated nitro groups is 2.64 Å.

There are hydrogen bonds between two water molecules and the O atoms of the 2,6-DNP ligands. The hydrogen bond length ranges from 2.664(4) [Ow2–Hw22···Ow3] to 3.092(4) Å [Ow3–Hw32···Ow2$^{\#1}$]. This means that the coherence of the hydrogen bond is weakened as the hydrogen bond length is increased.

As shown clearly at the coordination polyhedron of the La(III) ion in Fig. 2, the coordination environment of the La(III) center can be described as a bicapped square antiprism with two coordinated water molecules (Ow1 and Ow2) in the square planar position and eight oxygen donors (O1, O6, O11, and O11′ atoms from the phenol groups, and O2, O7, O12, and O15′ atoms from the nitro groups) of the chelating 2,6-DNP ligands. At relatively long distances, two O atoms (O7 and O15′) from nitro groups cap the upper and down sides of the square antiprism.

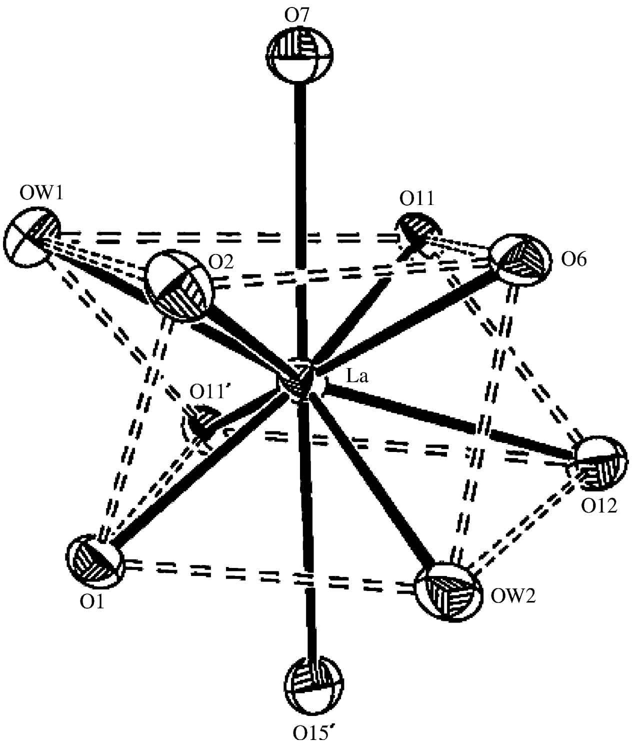

Fig. 2. The coordination polyhedron of the La atom in [La$_2$(2,6-DNP)$_6$(H$_2$O)$_4$]·4H$_2$O, **1**. The square antiprism is indicated by dashed lines.

The terbium 2,6-DNP complex appeared to be tri-hydrated neutral complex [Tb(2,6-DNP)$_3$(H$_2$O)$_3$], **2**. There are no water molecules in the outer sphere of the complex.

The selected bond lengths and angles for the complex **2** are listed in Table 3. An ORTEP diagram of the molecular structure for the complex **2** is presented by the atom numbering scheme in Fig. 3.

As shown in Fig. 3, the structural feature of complex **2** is the presence of three 2,6-DNP ligands coordinated in bidentate fashion directly to the Tb(III) atom. The structure is similar to 2,6-DNP complexes of Nd(III) and Y(III) [4,5] but different from that of La(III).

The Tb(III) ion is coordinated to nine O atoms. Three of these neighbors are the O atoms of water molecules (Ow1, Ow2, and Ow3). The Tb–O$_{\text{water}}$ bond distances are within

Table 3
Selected bond lengths (Å) and angles (°) for complex **2**

Tb—O1	2.279(5)	N2—O5	1.21(1)
Tb—Ow2	2.346(5)	N2—O4	1.243(9)
Tb—Ow3	2.401(7)	N2—C6	1.44(1)
Tb—Ow1	2.413(6)	O1—C1	1.281(9)
Tb—O4	2.607(6)	C1—C6	1.42(1)
O1—Tb—O4	66.2(2)	N2—O4—Tb	139.7(5)
Ow3—Tb—O7	140.3(2)	O1—C1—C6	126.9(7)
O4—N2—C6	119.5(7)	C1—C6—N2	120.4(7)
C1—O1—Tb	141.8(5)		

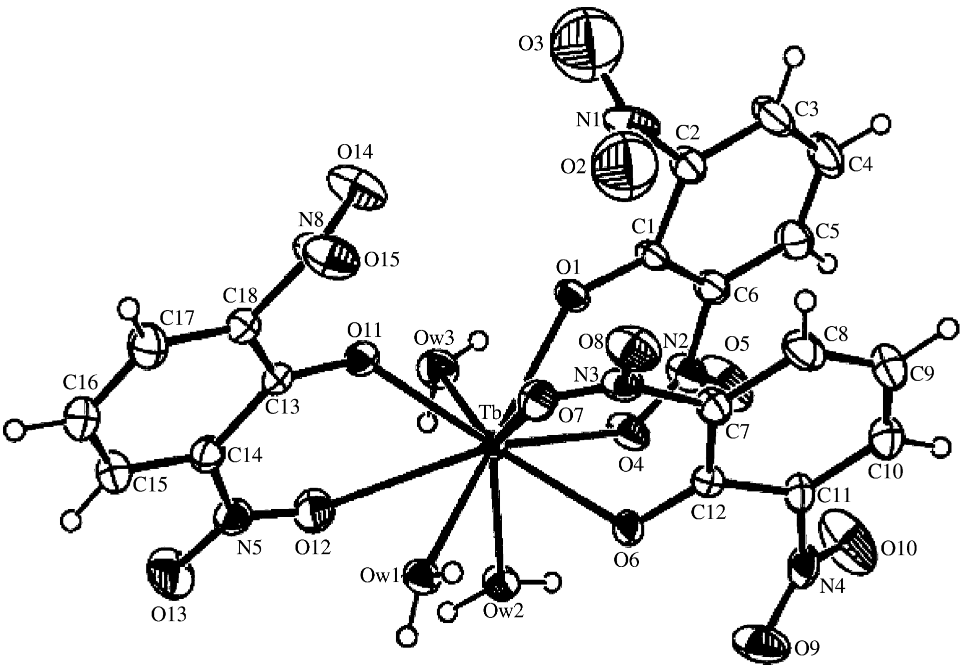

Fig. 3. The molecular structure of $[Tb(2,6\text{-}DNP)_3(H_2O)_3]$, **2**, showing the atom numbering scheme and 30% probability displacement ellipsoids.

the range of 2.346(5)–2.413(6) Å which are shorter than the sum of the covalent radii of the Tb and O atoms (2.42 Å). Tb(III) ion forms three six-membered chelate rings with each 2,6-DNP through an O atom of the nitro group and an O atom of the phenolate group.

The Tb–O distances to the nitro groups are in the range of 2.607(6)–2.721(7) Å, while the Tb–O distances to the phenolate groups are in the range of 2.247(5)–2.304(5) Å, showing that the bonds of Tb(III) ion to the O atoms of the nitro groups are significantly weak. However, the calculated dihedral angles between the benzene ring and the nitro groups are good evidence for an interaction between the O atoms on the nitro groups and the Tb atom. The dihedral angles between each benzene ring and its nitro groups are in the range 6.9(4)°–25.9(2)° for the coordinated nitro groups and 38.8(5)°–69.1(7)° for the uncoordinated nitro groups. The closest approach to Tb of the O atoms of the uncoordinated nitro groups is longer than 4 Å.

There are hydrogen bonds between three water molecules and the O atoms of the 2,6-DNP ligands. The hydrogen bond lengths range from 2.815(9) $[Ow2–Hw2B\cdots O15^{\#3}]$ to 3.15(2) Å $[Ow3–Hw3B\cdots O3^{\#4}]$.

Fig. 4 shows the coordination polyhedron of the Tb(III) ion in the complex **2**. The skeletal structure around the Tb atom forms a slightly distorted tricapped trigonal prism. Three water molecules coordinated directly to Tb(III) ion (Ow1, Ow2, and Ow3) and three oxygen atoms (O1, O6, and O11) from the phenolate groups of the 2,6-DNP ligands form a trigonal prism geometry occupying the basal sites. At relatively long distances, three O atoms (O4, O7, and O12) from the nitro groups of the ligands cap the sides of the trigonal prism.

3.2. Thermal analysis

DSC curve for the thermal decomposition of 2,6-DNP shows only two endothermic peaks, one sharp for the melting process at 64.0 °C and the other broad for the decomposition process at 254.8 °C [8].

The thermograms of TG-DTG and DSC of the $[La_2(2,6\text{-}DNP)_6(H_2O)_4]\cdot 4H_2O$, **1**, $Nd(2,6\text{-}DNP)_3(H_2O)_3$, $Tb(2,6\text{-}DNP)_3(H_2O)_3$, **2**, and $Y(2,6\text{-}DNP)_3(H_2O)_3$ were obtained under the conditions of linear temperature increase. The 2,6-DNP complexes of neodymium, terbium, and yttrium are all monomeric complexes whose chemical formulae are

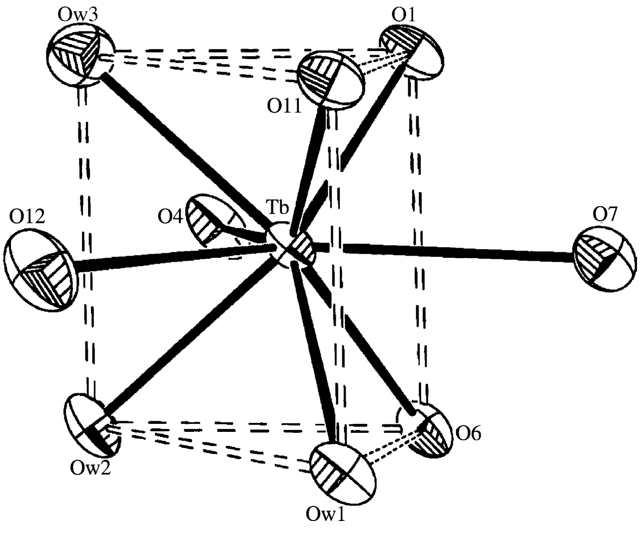

Fig. 4. The coordination polyhedron of the Tb atom in $[Tb(2,6\text{-}DNP)_3(H_2O)_3]$, **2**. The tricapped trigonal prism is indicated by dashed lines.

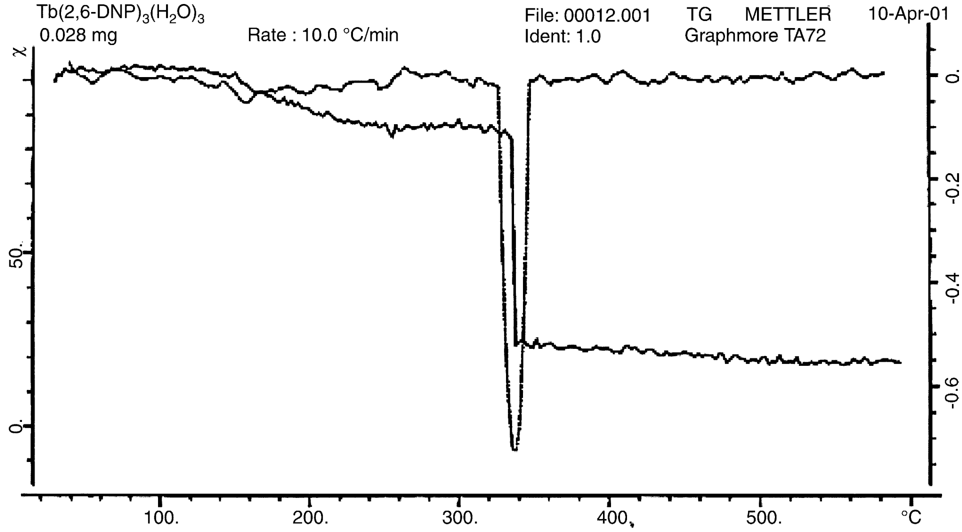

Fig. 5. TG-DTG curves of [Tb(2,6-DNP)$_3$(H$_2$O)$_3$]. Sample weight: 0.928 mg; atmosphere: nitrogen gas (20 mL/min, purity: 99.5%).

Ln(2,6-DNP)$_3$(H$_2$O)$_3$ (Ln = Nd, Tb, and Y), unlikely with the dimeric lanthanum complex **1**. However, the results of thermal analysis study for the lanthanide–2,6-DNP complexes are all similar each other in the patterns of TG-DTG and DSC curves. The major difference is that the dehydration temperatures (140–220 °C) for the monomeric complexes are about 80 °C higher than that for the dimeric complex **1**. The typical thermograms of TG-DTG and DSC of **2** are shown in Figs. 5 and 6, respectively. The thermal decomposition reaction of above complexes occurs through three stages under our experimental conditions, which are the dehydration, the ring breaking of 2,6-DNP ligand, and the formation of metal oxide.

The analytical details of TG-DTG and DSC curves for the terbium complex **2** are given here as an example. The dehydration step involves mass loss for the coordinated water molecules in the temperature range from 140 to 220 °C. In the process of dehydration for **2** to Tb(2,6-DNP)$_3$, the observed mass loss value of 7.4% is in good agreement with the calculated mass loss value of 7.1% corresponding to four water molecules dehydrated. The decomposition of Tb(2,6-DNP)$_3$ to Tb$_4$O$_7$ through the explosion of the 2,6-DNP coordinated occurs from 300 to 560 °C with the distinguished intermediate state around 340 °C. At present it could not be suggested what the intermediate would be. In previous paper for 3-nitro-1,2,4-triazol-5-one (NTO) complexes of lanthanide, it has been suggested that La$_2$(2,6-DNP)$_6$ would decompose thermally to La$_2$(CO$_3$)$_3$ from 300 to 460 °C and then to La$_2$O$_3$ from 460 to 560 °C [9].

It is assumed that the final stage would be the metal oxide formation of Tb$_4$O$_7$ from the intermediate in the temperature range of 340–560 °C. The observed mass loss value of 71% is in good agreement with the calculated value of 73.6% in this decomposition stage of Tb(2,6-DNP)$_3$ to Tb$_4$O$_7$.

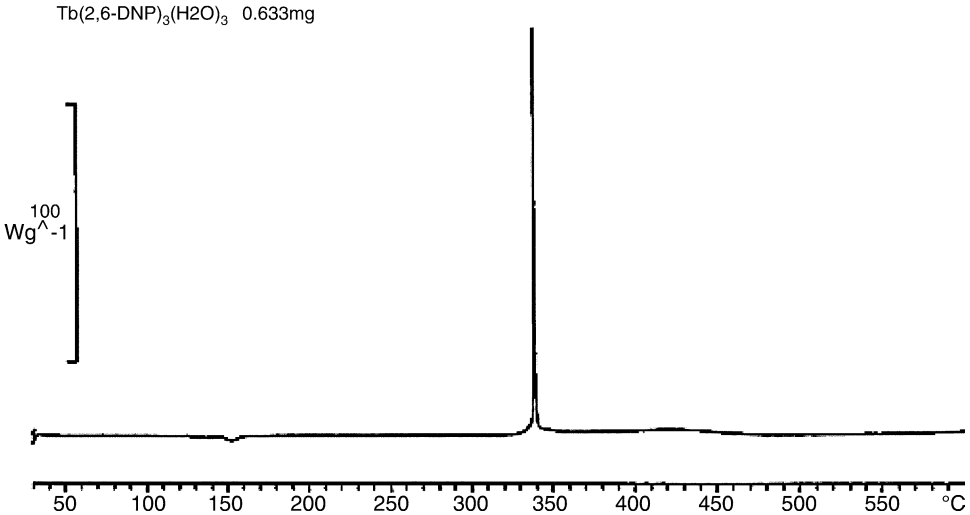

Fig. 6. DSC curve of [Tb(2,6-DNP)$_3$(H$_2$O)$_3$]. Sample weight: 0.663 mg; reference sample: indium (156.6 °C); atmosphere: static air.

On the other hand, the DSC curve shows again that there is only one significant exothermic peak at 340 °C which is identical with the peak temperature appeared on the DTG curve for the decomposition of Tb(2,6-DNP)$_3$. The endothermic peak corresponding to the dehydrations from the outer sphere in the temperature around 160 °C could not be found beside a small bump, since the endothermic energies for the dehydrations are so small to be appeared. It is interesting to see that the exothermic peak for the decomposition of coordinated 2,6-DNP becomes sharp and intense in narrower temperature range for the complex **2**. It would mean that the heat evolution by the decomposition is impactive possibly by the catalytic action of the metal ion. It was found from the DSC curves that the peak temperatures for explosive decompositions of La$_2$(2,6-DNP)$_6$, Nd(2,6-DNP)$_3$, Tb(2,6-DNP)$_3$, and Y(2,6-DNP)$_3$ are 342, 322, 340, and 343 °C, respectively.

Thus, the decomposition mechanism of Ln(2,6-DNP)$_3$ (Ln = La, Nd, Tb, and Y) would be summarized as:

$$[\mathrm{Ln(2,6\text{-}DNP)_3}] \xrightarrow{300\text{–}460\,^\circ\mathrm{C}} \text{intermediate mixture} \xrightarrow{460\text{–}560\,^\circ\mathrm{C}} \text{metal oxide.}$$

Acknowledgements

This work was supported by a grant (No. R01-2001-00055) from Korea Science and Engineering Foundation and a grant from Chungnam National University.

Appendix A. Supplementary

Complete lists with atomic coordinates, anisotropic displacement parameters, bond lengths and angles have been deposited at the Cambridge Crystallographic Data Center, 912 Union Road, Cambridge CB2 1EZ, UK [CCDC 242173 for La complex and CCDC 242174 for Tb complex].

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