Thermochimica Acta 490 (2009) 43–46

Contents lists available at ScienceDirect

Thermochimica Acta

journal homepage: www.elsevier.com/locate/tca

Thermochemical properties of the rare earth complexes with pyromellitic acid

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ARTICLE INFO

Article history:
Received 15 September 2008
Received in revised form 11 February 2009
Accepted 12 February 2009
Available online 24 February 2009

Keywords:
Rare earth complex
Thermal analysis
The enthalpy of combustion
The enthalpy of formation
Precise rotating-bomb calorimeter

ABSTRACT

Fourteen rare earth complexes with pyromellitic acid were synthesized and characterized by means of chemical and elemental analysis, and TG–DTG. The constant-volume combustion energies of complexes, $\Delta_c U$, were measured by a precise rotating-bomb calorimeter (RBC-type II). Their standard molar enthalpies of combustion, $\Delta_c H_m^\theta$, and standard molar enthalpies of formation, $\Delta_f H_m^\theta$, were calculated at $T = 298.15$ K. The relationship of $\Delta_c H_m^\theta$ and $\Delta_f H_m^\theta$ with the atomic numbers of the elements in the lanthanide series was examined. The results show that a certain amount of covalence is present in the chemical bond between rare earth cations and the ligand.

1. Introduction

In the past decades, the study of complexes involving with pyromellitic acid is a field of increasing interest [1–4]. Because pyromellitic acid possesses several interesting characteristics such as flexibility, versatile binding modes and ability to form hydrogen bonds [5], its complexes have fascinating coordination geometries and interesting structures, as well as the special properties [6–8]. Working along this line is motivated by the concept that these complexes have potential technological applications in magnetic and optical devices. Particularly, the rare earth complexes with pyromellitic acid have attracted the interest in functional materials science. Therefore, the number of reports concerning rare earth complexes constructed by pyromellitic acid has greatly increased [9–11].

Our research group joins in efforts on systematical investigation of the complexes' thermochemistry by employing a precise rotating-bomb calorimeter (RBC-type II) [12–14]. Previously we reported thermochemistry data of the rare earth complexes with salicylic acid [15]. Herein, following our systematic studies on salicylic acid containing rare earth complexes, we have undertaken the study of thermochemical properties of 14 rare earth complexes with pyromellitic acid. The TG–DTG of these complexes was examined. The constant-volume combustion energies, $\Delta_c U$, were determined. On the basis of $\Delta_c U$, their standard molar enthalpies of combustion, $\Delta_c H_m^\theta$, and standard molar enthalpies of formation, $\Delta_f H_m^\theta$, at $T = 298.15$ K were derived. Obviously, it is necessary to obtain basic thermodynamic data for theoretical researches and practical applications.

2. Experimental

2.1. Materials and instruments

Lanthanide chloride hydrate $RECl_3 \cdot nH_2O$ (RE = La–Lu, $n < 6$) was prepared according to Ref. [16]. 6.52 g (0.02 mol) La_2O_3 was dissolved in 20 cm^3 (6 mol dm^{-3}) hydrochloric acid, and the pH was adjusted to 4. Then the precipitate was formed by condensation under the reduced pressure. The reaction mixture was allowed to stand for 24 h. And it was filtered and laid in H_2SO_4 atmosphere. Finally, the products were dried by P_4O_{10} till constant weight. The cation content was determined by EDTA volumetric analysis, so the number of moles of water was indirectly derived from the molecular formulation, which includes the elemental analysis results. The product $LaCl_3 \cdot 6H_2O$ was 13.14 g, and the yield was 93%. The other compounds were prepared in the same way. Their purity is more than 99.9%.

Pyromellitic dianhydride of analytical reagent grade is commercially available from Tianjin Chemical Reagent Company. Sodium hydroxide and EDTA obtained from Xi'an Chemical Reagent Company are of analytical grade. Benzoic acid is purchased from Shanghai Reagent Company, with mass fraction purity 0.99999, and dried in a vacuum oven prior to use. All the chemicals are proved to be sufficiently reliable to be used without further purification.

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doi:10.1016/j.tca.2009.02.011

The C and H contents were measured by a Vario EL III elemental analyzer (Germany), and the constant-volume combustion energies of the complexes were determined with a precise rotating-bomb calorimeter (RBC-type II). The TG–DTG experiments were performed using a PerkinElmer thermogravimetric instrument.

2.2. Experimental conditions

The thermal analyses TG and DTG were carried out under a nitrogen atmosphere at a flow rate of 60 $cm^3\ min^{-1}$. The heating rate of 10 $K\ min^{-1}$ was chosen, with samples weight about 4–8 mg.

The main experimental procedures about RBC-type II were described previously [12]. This instrument was developed in our laboratory, and the calorimeter mainly consisted of a water thermostat, a caloritube, and the system for temperature measurement. The accuracy uncertainty was 4.18×10^{-4} by calibration with the standard benzoic acid. The bicyclic structure was used as the holder for the crucible support in the oxygen bomb, which facilitates the crucible stable relative to the bomb when the bomb was rotated in crosswise direction and vertically, assuring that the stable final state is attained in a short time.

The temperature of the water thermostat was maintained at $T = (298.1500 \pm 0.0005)$ K, and the initial oxygen pressure was 2.5 MPa. The correct value of the heat exchange was calculated according to *Linio–Pyfengdelel–Wsava* equation [17]. The energy equivalent of the calorimeter was calibrated with benzoic acid, which has an isothermal heat of combustion of $(-26434 \pm 3)\ J\ g^{-1}$ [18] at 298.15 K. The calibrated experimental results was $(18604.99 \pm 8.14)\ J\ K^{-1}$.

The analytical methods of final products (gas, liquid and solid) were the same as these in Ref. [12], and the analytical results of the final products indicated that the combustion reactions were complete. The methods of determination and calculation of the constant-volume combustion energies for complexes are similar to that for benzoic acid.

2.3. Preparation of complexes

6.54 g (0.03 mol) pyromellitic dianhydride was dissolved in NaOH solution. The molar ratio of pyromellitic dianhydride and sodium hydroxide is 1:2. The result solution was heated up in a water bath at 353 K. Then the compound $Na_2(H_2btec)$ was formed. 10.60 g (0.03 mol) $LaCl_3{\cdot}6H_2O$ was dissolved in distilled water (the molar ratio of $LaCl_3{\cdot}6H_2O$:Na_2H_2btec is 1:1), and the solution of $LaCl_3{\cdot}6H_2O$ was added dropwise into the solution of $Na_2(H_2btec)$ under electromagnetic stirring, and the precipitate was obtained. The reaction lasted for 2 h. After cooling the solution, the solid product was filtered and washed with distilled water and ethanol repeatedly. Then, it was dried in vacuum desiccator over P_4O_{10} till constant weight ready to be used. The product was 11.85 g, and the yield was 89%. As for other complexes, they were prepared the same as the pyromellitic acid complex of lanthanum. The purity of the complexes was 99.9%, which was checked by LC-10A type high performance liquid chromatography analyzer (made in Japan), adopting the solvent DMF, the mobile phase DMF–H_2O and the reversed phase C_{18} column.

3. Results and discussion

3.1. Composition of the solid complexes

The analytical results of the composition of these complexes are presented in Table 1. RE^{3+} was determined by EDTA volumetric analysis. Based on the data, the composition of these complexes can be identified with the general formula RE(Hbtec)·$n$$H_2O$ (Hbtec = $C_{10}H_3O_8{}^{3-}$; RE = La–Nd, Tb–Lu, $n = 3$; RE = Sm–Gd, $n = 2$).

Table 1
Analytical results related to the composition of the complexes.

Complex	*w* (found) (%) (*w* (calculated) (%))			Yield (%)
	RE	C	H	
La(Hbtec)·$3H_2O$	31.07 (31.28)	26.89(27.05)	1.91(2.04)	89
Ce(Hbtec)·$3H_2O$	31.40(31.47)	26.84(26.97)	2.07(2.04)	86
Pr(Hbtec)·$3H_2O$	31.52(31.59)	26.89(26.93)	1.95(2.03)	85
Nd(Hbtec)·$3H_2O$	32.21(32.10)	26.65(26.73)	1.93(2.02)	87
Sm(Hbtec)·$2H_2O$	34.25(34.37)	27.36(27.45)	1.71(1.61)	81
Eu(Hbtec)·$2H_2O$	34.40(34.61)	27.23(27.35)	1.69(1.61)	83
Gd(Hbtec)·$2H_2O$	35.17(35.38)	26.74(27.03)	1.52(1.59)	81
Tb(Hbtec)·$3H_2O$	34.35(34.24)	25.78(25.88)	1.87(1.95)	75
Dy(Hbtec)·$3H_2O$	34.88(34.75)	25.79(25.68)	2.01(1.94)	77
Ho(Hbtec)·$3H_2O$	35.17(35.08)	25.64(25.55)	1.87(1.93)	78
Er(Hbtec)·$3H_2O$	35.56(35.40)	25.49(25.42)	1.95(1.92)	73
Tm(Hbtec)·$3H_2O$	35.49(35.63)	25.42(25.33)	1.88(1.91)	71
Yb(Hbtec)·$3H_2O$	36.31(36.19)	25.20(25.12)	1.85(1.90)	71
Lu(Hbtec)·$3H_2O$	36.37(36.44)	24.91(25.02)	1.93(1.89)	69

3.2. TG–DTG of the solid complexes

The thermal decompositional procedures of La and Er complexes are presented in Figs. 1 and 2. As for other complexes, the TG–DTG figures are similar with La complex or Er complex, respectively. As shown in TG–DTG curves, the thermal decomposition process of Er, Tm, Yb and Lu complexes with pyromellitic acid is

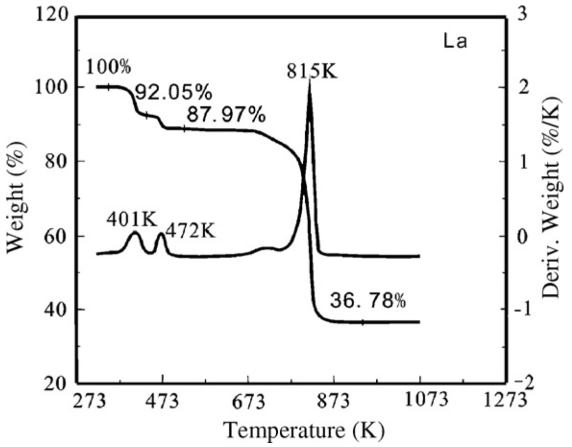

Figure 1. TG–DTG curves of La complex.

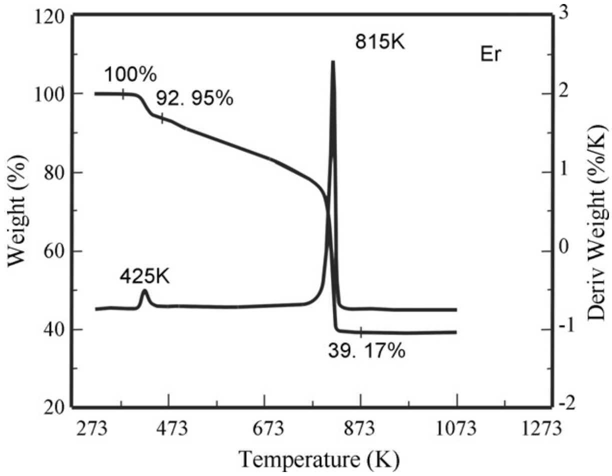

Figure 2. TG–DTG curves of Er complex.

Table 2
TG data of rare earth complexes with pyromellitic acid.

RE	$RE(Hbtec){\cdot}H_2O$			$RE(Hbtec)$			$(1/2)RE_2O_3$ (Ce is the CeO_2)		
	Temperature range (K)	Residue (%)		Temperature range (K)	Residue (%)		Temperature range (K)	Residue (%)	
		Found	Calculated		Found	Calculated		Found	Calculated
La	298–401	92.05	91.88	401–472	87.97	87.83	472–815	36.78	36.68
Ce	298–383	91.78	91.91	383–465	87.53	87.86	465–733	36.95	38.65
Pr	298–379	91.83	91.92	379–450	87.79	87.88	450–789	36.88	36.97
Nd	298–375	91.80	91.98	375–451	87.91	87.97	451–811	37.32	37.44
Sm	298–420	96.01	95.88	420–482	91.65	91.76	482–798	39.85	39.85
Eu	298–396	95.75	95.90	396–489	91.83	91.79	489–766	40.64	40.18
Gd	298–412	96.03	95.95	412–493	92.23	91.89	493–807	41.63	40.78
Tb	298–394	92.40	92.24	394–496	88.41	88.35	496–803	39.95	39.42
Dy	298–390	92.45	92.30	390–544	88.51	88.44	544–815	40.58	39.88
Ho	298–378	92.25	92.34	378–544	88.13	88.50	544–815	40.02	40.19
Er	298–425	92.95	92.37	–	–	–	425–815	39.17	40.48
Tm	298–422	92.29	92.40	–	–	–	422–804	40.57	40.69
Yb	298–419	92.43	92.46	–	–	–	419–799	41.08	41.20
Lu	298–413	92.36	92.50	–	–	–	413–812	41.33	41.44

divided into two stages, while that of the other complexes takes three steps. However, the products of the first intermediate stage and the final decomposition of all complexes are similar, which are $RE(Hbtec){\cdot}H_2O$ and (1/2) RE_2O_3 (Ce is the CeO_2), respectively. Moreover, The intermediate products of the second stage of the complexes with three steps are RE(Hbtec).

In addition, it can be also seen that the temperature of the first decomposition stage is in the temperature range of 375–425 K, all the complexes have the dehydration peaks. In the second stage, $RE(Hbtec){\cdot}nH_2O$ (form La to Ho) still have the dehydration peaks, but the dehydration peaks in heavy rare earth complexes are special that their TG curves have no platform after complete dehydration. The reason is probably that the lanthanide atomic radius shrinks, so the metal ion with big radius can exist stably after dehydrating, while the heavy rare earth complex with small radius dehydrates and decomposes at the same time. Therefore, it can be concluded that from Er to Lu hydrated heavy rare earth complexes are less stable than light and middle rare earth complexes. The range of the temperature in the last decomposition stage is from 733 K to 815 K. The residue of in every step of 14 complexes with pyromellitic acid is presented in Table 2. The temperature of CeO_2 formation is lowest. That is probably because the intermediate product of Ce(III) converts into Ce(IV).

3.3. *Constant-volume combustion energies of the complexes*

The experimental results of the constant-volume combustion energies of complexes are summarized in Table 3. The values were calculated by the following formula:

$$-\Delta_c U = \frac{W\Delta T - Gb - 5.97c}{a} \tag{1}$$

where $\Delta_c U$ (complexes, s) denotes the constant-volume combustion energy of the complexes (in $J\,g^{-1}$), W the energy equivalent of the rotating-bomb calorimeter (in $J\,K^{-1}$), a is the mass (in g) of the complex, b is the length of the actual Ni–Cr wire consumed (in cm). G is the combustion enthalpy of Ni–Cr wire for ignition ($0.9\,J\,cm^{-1}$), 5.97 the formation enthalpy and solution enthalpy of nitric acid corresponding to $1\,cm^3$ of $0.1000\,mol\,dm^{-3}$ NaOH (in $J\,cm^{-3}$), c is the volume (in cm^3) of consumed $0.1000\,mol\,dm^{-3}$ NaOH and ΔT is the correct value of the temperature rise. The constant-volume combustion energy of each complex is repetitively determined six times.

Table 3
The constant-volume combustion energies, standard molar enthalpies of combustion and standard molar enthalpies of formation for the complexes.

Complexes	$\Delta_c U$ ($kJ\,mol^{-1}$)	$\Delta_c H_m^\theta$ ($kJ\,mol^{-1}$)	$\Delta_f H_m^\theta$ ($kJ\,mol^{-1}$)
$La(Hbtec){\cdot}3H_2O$	-3609.67 ± 0.84	-3603.47 ± 0.84	-2513.67 ± 22.24
$Ce(Hbtec){\cdot}3H_2O$	-3314.90 ± 1.03	-3309.32 ± 1.03	-3002.42 ± 22.25
$Pr(Hbtec){\cdot}3H_2O$	-3901.00 ± 1.17	-3894.05 ± 1.17	-2232.24 ± 22.28
$Nd(Hbtec){\cdot}3H_2O$	-3548.99 ± 0.99	-3542.79 ± 0.99	-2582.00 ± 22.28
$Sm(Hbtec){\cdot}2H_2O$	-3409.43 ± 0.77	-3403.23 ± 0.77	-2445.68 ± 19.27
$Eu(Hbtec){\cdot}2H_2O$	-3930.45 ± 0.66	-3924.25 ± 0.66	-1842.51 ± 19.35
$Gd(Hbtec){\cdot}2H_2O$	-3504.61 ± 0.84	-3498.41 ± 0.84	-2346.95 ± 19.21
$Tb(Hbtec){\cdot}3H_2O$	-3885.69 ± 0.95	-3879.49 ± 0.95	-2274.45 ± 22.43
$Dy(Hbtec){\cdot}3H_2O$	-3942.97 ± 0.96	-3936.77 ± 0.96	-2216.27 ± 22.36
$Ho(Hbtec){\cdot}3H_2O$	-3794.10 ± 1.28	-3787.90 ± 1.28	-2375.09 ± 22.62
$Er(Hbtec){\cdot}3H_2O$	-4041.62 ± 0.66	-4035.42 ± 0.66	-2135.97 ± 22.45
$Tm(Hbtec){\cdot}3H_2O$	-3995.30 ± 0.85	-3989.10 ± 0.85	-2176.89 ± 22.40
$Yb(Hbtec){\cdot}3H_2O$	-3457.64 ± 0.84	-3451.44 ± 0.84	-2677.15 ± 22.42
$Lu(Hbtec){\cdot}3H_2O$	-3441.56 ± 0.99	-3435.36 ± 0.99	-2724.48 ± 22.56

3.4. *Standard molar combustion enthalpies of the complexes*

The standard molar combustion enthalpies of the complexes, $\Delta_c H_m^\theta$, refer to the combustion enthalpy changes of the following ideal combustion reaction at 298.15 K and 101.325 kPa:

$$\begin{aligned} &RE(C_{10}H_3O_8){\bullet}3H_2O(s) + (15/2)O_2(g) \\ &\quad = (1/2)La_2O_3(s) + 10CO_2(g) + (9/2)H_2O(l) \\ &\qquad (RE = \text{La–Nd, Tb–Lu, except for Ce}) \end{aligned} \tag{A}$$

$$\begin{aligned} &Ce(C_{10}H_3O_8){\bullet}3H_2O(s) + (31/4)O_2(g) \\ &\quad = CeO_2(s) + 10CO_2(g) + (9/2)H_2O(l) \end{aligned} \tag{B}$$

$$\begin{aligned} &RE(C_{10}H_3O_8){\bullet}2H_2O(s) + (15/2)O_2(g) \\ &\quad = (1/2)RE_2O_3(s) + 10CO_2(g) + (7/2)H_2O(l) \\ &\quad (RE = \text{Sm–Gd}) \end{aligned} \tag{C}$$

The standard molar combustion enthalpies of the complexes are calculated by the following equations:

$$\Delta_c H_m^\theta = \Delta_c U + \Delta nRT \tag{2}$$

$$\Delta n = n_g(\text{products}) - n_g(\text{reactants})$$

where n_g is the total amount (in mol) of gases present as products or as reactants, $R = 8.314\,J\,mol^{-1}\,K^{-1}$, $T = 298.15$ K. The results of the calculation are given in Table 3.

Table 4
The standard molar enthalpies of formation of the rare earth oxide, $H_2O(l)$ and $CO_2(g)$.

Rare earth oxide	$\Delta_f H_m^\theta$ (kJ mol^{-1})	Rare earth oxide	$\Delta_f H_m^\theta$ (kJ mol^{-1})
$La_2O_3(s)$	−1791.6 ± 2.0	CeO_2 (s)	−1090.4 ± 1.0
$Pr_2O_3(s)$	−1809.9 ± 3.0	Nd_2O_3 (s)	−1806.9 ± 3.0
$Sm_2O_3(s)$	−1826.8 ± 4.8	Eu_2O_3 (s)	−1662.5 ± 6.0
$Gd_2O_3(s)$	−1819.7 ± 3.6	Tb_2O_3 (s)	−1865.2 ± 6.0
$Dy_2O_3(s)$	−1863.4 ± 5.0	Ho_2O_3 (s)	−1883.3 ± 8.2
$Er_2O_3(s)$	−1900.1 ± 6.5	Tm_2O_3 (s)	−1889.3 ± 5.7
$Yb_2O_3(s)$	−1814.5 ± 6.0	Lu_2O_3 (s)	−1877.0 ± 7.7
$CO_2(g)$	−393.51 ± 1.3	H_2O (l)	−285.830 ± 4.0

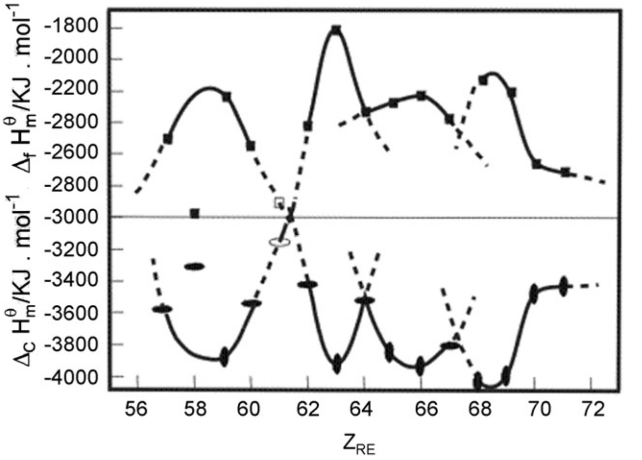

Figure 3. Plot of and values against the atomic numbers of the rare earths.

3.5. Standard molar enthalpies of formation for the complexes

The standard molar enthalpies of formation of the compounds, $\Delta_f H_m^\theta$, are calculated by Hess's law according to the thermochemical Eqs. (3)–(5), respectively:

$$\Delta_f H_m^\theta(s) = \frac{1}{2}\Delta_f H_m^\theta(RE_2O_3, s) + 10\Delta_f H_m^\theta(CO_2, g) + \frac{9}{2}\Delta_f H_m^\theta(H_2O, l) - \Delta_c H_m^\theta(RE(C_{10}H_3O_8)\cdot 3H_2O, s) \quad (RE = La\text{–}Nd,\ Tb\text{–}Lu,\ \text{except for Ce}) \tag{3}$$

$$\Delta_f H_m^\theta(s) = \Delta_f H_m^\theta(CeO_2, s) + 10\Delta_f H_m^\theta(CO_2, g) + \frac{9}{2}\Delta_f H_m^\theta(H_2O, l) - \Delta_c H_m^\theta(Ce(C_{10}H_3O_8)\cdot 3H_2O, s) \tag{4}$$

$$\Delta_f H_m^\theta(s) = \frac{1}{2}\Delta_f H_m^\theta(RE_2O_3, s) + 10\Delta_f H_m^\theta(CO_2, g) + \frac{7}{2}\Delta_f H_m^\theta(H_2O, l) - \Delta_c H_m^\theta(RE(C_{10}H_3O_8)\cdot 2H_2O, s) \quad (RE = Sm\text{–}Gd) \tag{5}$$

The standard molar enthalpies of formation of the rare earth oxide, $H_2O(l)$ and $CO_2(g)$ were obtained from Refs. [19,20] were listed in Table 4. The results of the calculations are also listed in Table 3.

In Fig. 3, we have given that $\Delta_c H_m^\theta$, $\Delta_f H_m^\theta$ of the above complexes are plotted against the atomic numbers of the elements in the lanthanide series. The curve shows the "tetrad effect" of rare earth, suggesting that a certain amount of covalence is present in the chemical bonds between RE^{3+} and ligand, which is caused by the incomplete shield of $5s^25p^6$ orbital to 4f electrons. The experimental result is in agreement with the Nephelauxetic effect of 4f electrons of rare earth.

On the basis of Fig. 3, the corresponding standard enthalpy of combustion and the standard enthalpy of formation of $Pm(Hbtec)\cdot 3H_2O$ could be estimated as being −3150 kJ mol^{-1} and −2910 kJ mol^{-1}, respectively.

Acknowledgements

The investigation received financial assistance from Shaanxi Province of China (Grant No. 2007K02-10), Education Committee of Shaanxi Province (Grant No. 05JC31).

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