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Standard molar enthalpies of formation of $Ln(C_9H_6NO)_2(C_2H_3O_2)$ (Ln: Nd, Sm)

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Abstract

Using an on-line solution-reaction isoperibol calorimeter, the standard molar enthalpies of reaction for the general thermochemical reaction: $LnCl_3 \cdot 6H_2O(s) + 2C_9H_7NO(s) + CH_3COONa(s) = Ln(C_9H_6NO)_2(C_2H_3O_2)(s) + NaCl(s) + 2HCl(g) + 6H_2O(l)$ (Ln: Nd, Sm), were determined at $T = 298.15$ K, as $-(56.9 \pm 1.4)$ and $-(67.9 \pm 1.5)$ kJ mol^{-1}, respectively. From the mentioned standard molar enthalpies of reaction and other auxiliary thermodynamic quantities, the standard molar enthalpies of formation of $Ln(C_9H_6NO)_2(C_2H_3O_2)(s)$ (Ln: Nd, Sm), at $T = 298.15$ K, have been derived to be: $-(1494.7 \pm 3.3)$ and $-(1501.5 \pm 3.4)$ kJ mol^{-1}, respectively.
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Keywords: Solution-reaction calorimetry; Standard enthalpy of formation; Coordination compound of rare earth; 8-Hydroxyquinoline; Acetic acid

1. Introduction

Thermochemical data for metal complexes of 8-hydroxyquinoline were firstly published by Jamea and Pilcher in 1985 [1]. The standard molar enthalpies of formation and sublimation of some crystalline bis-(8-hydroxyquinoline) and bis-(8-hydroxy-2-methylquinoline) metal complexes have been determined by Ribeiro da Silva and his collaborators in 1994 [2] and 1998 [3], respectively. In our previous studies, Qu and co-workers have measured the values of the standard molar enthalpies of formation of some metal complexes of 8-hydroxyquinoline [4,5]. As part of our interests in the thermochemistry of complexes of rare earth, we have recently reported thermodynamic studies of reactions of rare earths with 8-hydroxyquinoline [6,7]. Thermochemical data are needed to help understanding the driving forces in the seemingly complex reactivity displayed in coordination chemistry. The energetics associated with it is of importance in the formation of coordination compounds. Despite that, there is relatively little reliable experimental data available for these coordination compounds. For this reason, a systematic study of thermodynamics of the coordination compounds was performed by us.

In the present study, the standard molar enthalpies of reaction for the general thermochemical reaction, $LnCl_3 \cdot 6H_2O(s) + 2C_9H_7NO(s) + CH_3COONa(s) = Ln(C_9H_6NO)_2(C_2H_3O_2)(s) + NaCl(s) + 2HCl(g) + 6H_2O(l)$ (Ln: Nd, Sm), were determined by solution-reaction calorimetry and the standard molar enthalpies of formation of $Ln(C_9H_6NO)_2(C_2H_3O_2)$ (Ln: Nd, Sm) were derived.

2. Experimental

2.1. Chemicals

Potassium chloride KCl (mass fraction 0.9999) and sodium chloride NaCl (mass fraction 0.9999) were purchased from Shanghai No. 1 Reagent Factory, Shanghai, PR China. They were dried in a vacuum oven at 500 K for 6 h prior to use. The chemicals, neodymium(III) chloride hexahydrate NdCl$_3$·6H$_2$O (mass fraction 0.995), samarium(III) chloride hexahydrate SmCl$_3$·6H$_2$O (mass fraction 0.995), sodium acetate CH$_3$COONa (NaAc; mass fraction 0.999) and 8-hydroxyquinoline C$_9$H$_7$NO (abbreviated as Hhq, mass fraction higher than 0.995) were commercially

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Table 1
Infrared bonds (cm^{-1}) for 8-hydroquinoline, CH_3COONa and $Ln(hq)_2Ac$ (Ln: Nd, Sm)

Compound	ν (OH)	δ (OH)	ν (C=N)	ν (C–O)	ν_{as} (COO)	ν_s (COO)	$\Delta\nu^a$	ν (Ln–N)
Hhq	3090	1028	1579	1094				
NaAc					1561	1412	149	
Nd(hq)$_2$Ac			1572	1105	1498	1465	33	486
Sm(hq)$_2$Ac			1571	1105	1498	1464	34	486

^{a} $\Delta\nu = \nu_{as}(COO) - \nu_s(COO)$.

obtained from Shanghai Chemical Reagent Co., Shanghai, PR China. They were maintained in desiccators over phosphorus pentoxide (P_2O_5) or silica gel before the calorimetric measurements. Hydrochloric acid (analytical grade) and double-distilled water were used to prepare all of the calorimetric solvents.

2.2. Synthesis and characterisation of the coordination compounds

The synthesis and purification of $Ln(hq)_2Ac$ were carried out as previously described in the literature [8]. $LnCl_3·6H_2O$ (Ln: Nd or Sm), an equivalent amount of NaAc with two equivalents of Hhq reacted in mixing solution of alcohol and double-distilled water (5:1) at 330 K for 3–4 h (pH = 7–8). The yellow precipitates were filtered, and the products were subsequently washed with double-distilled water and then absolute alcohol for three times, respectively, and further dried in a vacuum oven at 330 K for 24 h.

The FT-IR spectra of the two compounds in the range of 4000–400 cm^{-1} were obtained from KBr pellets using a FT-IR spectrophotometer (Model Avatar 360, Thermo Nicolet, USA). As can be observed in Table 1, the atoms of rare earth are chelated by the nitrogen and oxygen atom of 8-hydroxyquinoline. The synthesized coordination compounds exhibit a negative shift of the antisymmetric and a positive shift of the symmetric stretching mode vibrations of COO^- group, in comparison to NaAc. The contents of rare earth in the two coordination compounds were determined by EDTA titration, and the element carbon, nitrogen and hydrogen of the two compounds were checked by an elemental analyzer (Model 1106, Carlo Erba Strumentazione, Italy), which were agreement with the proposed formula established for the two compounds, as shown in Table 2. The TG-DTA tests were performed in a thermal analyzer (Model Setsys 16/18, Setaram, France) and under dynamic atmosphere of high purity N_2 (mass fraction 0.99999) with a

flow rate of 60 cm^3 min^{-1} and a heating rate of 10 K min^{-1}. The two coordination compounds were very stable, and the thermal decomposition temperature of them was above 620 K.

2.3. Solution-reaction isoperibol calorimeter

An on-line solution-reaction isoperibol calorimeter constructed in the laboratory was used to determine the enthalpy of dissolution. The calorimeter consisted of a water thermostat, a pyrex-glass Dewar, a glass sample cell, a glass-sheathed thermistor probe and precision Wheatstone bridge, a heater for electric energy calibration and equilibration purposes, a stabilized current supply, a digital electronic timer, a data acquisition/switch unit, a 20-channel multiplexer module, a multifunction module (Model 34970A, 34901A and 34907A, Agilent, USA), and a personal computer for data acquisition and processing. During the experiments, the water thermostat was maintained at $T = (298.15 \pm 0.001)$ K, and the resolution of temperature measurement was $\pm 1 \times 10^{-4}$ K. The inevitable heat transfer and heat generation due to friction were compensated and the adiabatic temperature change (the corrected voltage change) was automatically calculated by the program solution-reaction calorimetry system (SRCS) complied by us using the equal area method. More comprehensive description and detailed procedure of the calorimeter can be found in the literature [7].

In order to verify the reliability of the calorimeter, the molar enthalpy of solution of KCl (Standard Reference Material 1655, the National Institute of Standards and Technology) in double-distilled water was measured at $T = 298.15$ K. The molar enthalpy of solution of KCl in double-distilled water was (17560 ± 21) J mol^{-1}, which was in good agreement with the published values in the literatures [9,10]. The uncertainty and the inaccuracy of the experimental results were within $\pm 0.3\%$ compared with the recommended reference data.

2.4. Determination of the enthalpies of dissolution

The general thermochemical reaction for measuring the standard molar enthalpies of reaction was depicted in Fig. 1.

Samples of 0.5 mmol [NdCl$_3$·6H$_2$O(s), 2Hhq(s) and NaAc(s)] were dissolved in 100.0 cm^3 4.0 mol dm^{-3}

Table 2
Percentages of experimental (calculated) values for elemental analysis of $Ln(hq)_2Ac$ (Ln: Nd, Sm)

Compound	Ln (%)	C (%)	N (%)	H (%)
Nd(hq)$_2$Ac	29.2 (29.3)	48.7 (48.9)	5.8 (5.7)	3.1 (3.1)
Sm(hq)$_2$Ac	30.2 (30.2)	48.2 (48.3)	5.6 (5.6)	3.1 (3.0)

$$\text{LnCl}_3 \cdot 6\text{H}_2\text{O(s)} + 2\text{Hhq(s)} + \text{NaAc(s)} \xrightarrow{\Delta_r H_m^{\Theta}} 2\text{HCl(g)} + 6\text{H}_2\text{O(l)} + \text{Ln(hq)}_2\text{Ac(s)} + \text{NaCl(s)}$$

$$\Delta_s H_m^{\Theta} \qquad\qquad \Delta_s H_m^{\Theta} \qquad\qquad \Delta_s H_m^{\Theta}$$
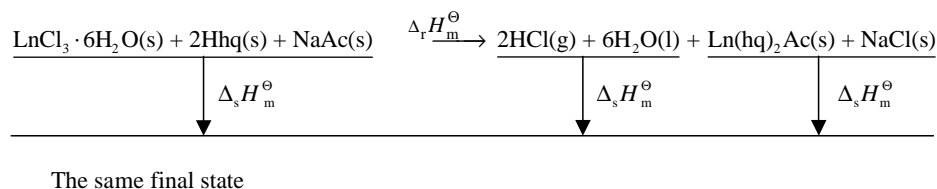
The same final state

Fig. 1. The general thermochemical cycles for measuring the molar enthalpies of reaction, for Nd is the reaction (1) and for Sm is the reaction (2).

Table 3
The molar enthalpy of dissolution of 0.5 mmol [NdCl$_3$·6H$_2$O(s), 2Hhq(s) and NaAc(s)] in 100.0 cm^3 4.0 mol dm^{-3} HCl(aq) at $T = 298.15$ K[b]

| No.	m (g)			Q_s (J)	$\Delta_s H_m^{\Theta}$ (kJ mol^{-1})
	NdCl$_3$·6H$_2$O (s)	Hhq (s)	NaAc (s)		
1	0.1795	0.1451	0.0407	−24.49	−48.97
2	0.1794	0.1451	0.0413	−23.71	−47.42
3	0.1794	0.1455	0.0410	−23.86	−47.71
4	0.1795	0.1453	0.0409	−23.54	−47.08
5	0.1791	0.1455	0.0409	−23.57	−47.13
Mean					−(47.66 ± 0.77)

[b] Q_s is the heat of dissolution; $\Delta_s H_m^{\Theta}$ is the molar enthalpy of dissolution, and the uncertainty quoted for $\Delta_s H_m^{\Theta}$ corresponded to the standard deviation.

HCl(aq) at $T = 298.15$ K.

$$\{\text{NdCl}_3 \cdot 6\text{H}_2\text{O(s)} + 2\text{Hhq(s)} + \text{NaAc(s)}\} + S_1$$
$$\rightarrow \text{solution A} \tag{3}$$
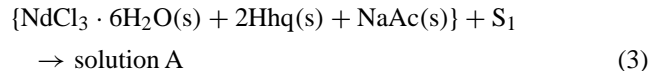
where S_1 was the calorimetric solvent.

Samples of 0.5 mmol [Nd(hq)$_2$Ac(s) and NaCl(s)] were dissolved in [100.0 cm^3 4.0 mol dm^{-3}HCl(aq) + 1.0 mmol HCl + 3.0 mmol H$_2$O] at $T = 298.15$ K.

$$\{\text{Nd(hq)}_2\text{Ac(s)} + \text{NaCl(s)}\} + S_2 \rightarrow \text{solution B} \tag{4}$$
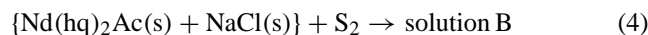
where S_2 was the calorimetric solvent.

The calorimetric results of reactions (3) and (4) were listed in Tables 3 and 4. Samples of 0.5 mmol [SmCl$_3$·6H$_2$O(s), 2Hhq(s) and NaAc(s)] were dissolved in 100.0 cm^3 4.0 mol dm^{-3} HCl(aq) at $T = 298.15$ K

Table 4
The molar enthalpy of dissolution of 0.5 mmol [Nd(hq)$_2$Ac(s) and NaCl(s)] in [100.0 cm^3 4.0 mol dm^{-3} HCl(aq) + 1.0 mmol HCl + 3.0 mmol H$_2$O] at $T = 298.15$ K

No.	m (g)		Q_s (J)	$\Delta_s H_m^{\Theta}$ (kJ mol^{-1})
	Nd(hq)$_2$Ac (s)	NaCl (s)		
1	0.2461	0.0294	−66.56	−132.96
2	0.2461	0.0295	−65.79	−131.42
3	0.2460	0.0291	−65.52	−130.94
4	0.2458	0.0294	−66.07	−132.13
5	0.2460	0.0291	−65.02	−129.93
Mean				−(131.48 ± 1.15)

Table 5
The molar enthalpy of dissolution of 0.5 mmol [SmCl$_3$·6H$_2$O(s), 2Hhq(s) and NaAc(s)] in 100.0 cm^3 4.0 mol dm^{-3} HCl(aq) at $T = 298.15$ K

No.	m (g)			Q_s (J)	$\Delta_s H_m^{\Theta}$ (kJ mol^{-1})
	SmCl$_3$·6H$_2$O (s)	Hhq(s)	NaAc (s)		
1	0.1822	0.1452	0.0409	−22.50	−45.15
2	0.1826	0.1453	0.0410	−22.21	−44.38
3	0.1825	0.1454	0.0412	−22.10	−44.18
4	0.1827	0.1454	0.0412	−22.08	−44.10
5	0.1823	0.1454	0.0413	−22.10	−44.23
Mean					−(44.41 ± 0.43)

Table 6
The molar enthalpy of dissolution of 0.5 mmol [Sm(hq)$_2$Ac(s) and NaCl(s)] in [100.0 cm^3 4.0 mol dm^{-3} HCl(aq) + 1.0 mmol HCl + 3.0 mmol H$_2$O] at $T = 298.15$ K

No.	m (g)		Q_s (J)	$\Delta_s H_m^{\Theta}$ (kJ mol^{-1})
	Sm(hq)$_2$Ac(s)	NaCl(s)		
1	0.2487	0.0290	−59.37	−118.81
2	0.2488	0.0294	−59.28	−118.59
3	0.2489	0.0291	−57.86	−115.70
4	0.2488	0.0296	−58.67	−117.36
5	0.2487	0.0295	−57.98	−116.03
Mean				−(117.30 ± 1.42)

$$\{\text{SmCl}_3 \cdot 6\text{H}_2\text{O(s)} + 2\text{Hhq(s)} + \text{NaAc(s)}\} + S_1$$
$$\rightarrow \text{solution C} \tag{5}$$
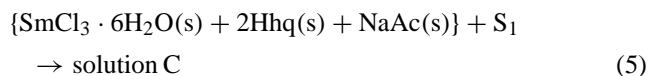
where S_1 was the calorimetric solvent.

Samples of 0.5 mmol [Sm(hq)$_2$Ac(s) and NaCl(s)] were dissolved in [100.0 cm^3 4.0 mol dm^{-3} HCl(aq) + 1.0 mmol HCl + 3.0 mmol H$_2$O] at $T = 298.15$ K.

$$\{\text{Sm(hq)}_2\text{Ac(s)} + \text{NaCl(s)}\} + S_2 \rightarrow \text{solution D} \tag{6}$$
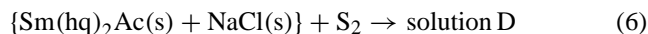
where S_2 was the calorimetric solvent. The calorimetric results of reactions (5) and (6) were showed in Tables 5 and 6.

3. Results and discussion

The standard atomic masses recommended by the IUPAC Commission in 2001 [11] were used in the calculation of all molar quantities.

3.1. Evaluation of the enthalpy of dissolution

The molar enthalpy of dissolution of [2.0 mol HCl(g) + 6.0 mol H$_2$O(l)] in [2000 × 100.0 cm^3] 4.0 mol dm^{-3} HCl(aq) at $T = 298.15$ K was calculated.

$$\Delta_s H_m^\Theta(7) = 2\Delta_s H_\infty^\Theta[\text{HCL(g)}]$$
$$- 2\Delta_d H_\infty^\Theta[\text{HCL(aq, 4.000} \to 0]$$
$$= 2[74.772 - 4.397] = 140.750$$

3.2. Calculation of the standard molar enthalpy of reaction

$$\Delta_r H_m^\Theta(1) = \Delta_s H_m^\Theta(3) - \Delta_s H_m^\Theta(4) - \Delta_s H_m^\Theta(7)$$
$$= [-47.66] - [-131.48] - [140.750]$$
$$- (56.9 \pm 1.4)$$

$$\Delta_r H_m^\Theta(2) = \Delta_s H_m^\Theta(5) - \Delta_s H_m^\Theta(6) - \Delta_s H_m^\Theta(7)$$
$$= [-44.41] - [-117.30] - [140.750]$$
$$= -(67.9 \pm 1.5)$$

The uncertainties, s, assigned to $\Delta_r H_m^\Theta$, were obtained considering the uncertainties of $\Delta_s H_m^\Theta$ and calculated from the standard deviations:

$$s\,\Delta_r H_m^\Theta = \left\{\sum [s\,\Delta_s H_m^\Theta]^2\right\}^{1/2}$$

where $s\,\Delta_s H_m^\Theta$ was the corresponding uncertainty of the standard molar enthalpy of dissolution.

The standard molar enthalpies of reaction for the two reactions were tabulated in Table 7.

3.3. Derivation of the standard molar enthalpy of formation

From the standard molar enthalpies of reaction for the two reactions and various ancillary data listed in Table 8, the standard molar enthalpies of formation of the two coordination compounds were calculated.

$$\Delta_f H_m^\Theta[\text{Nd(hq)}_2\text{Ac(s)}]$$
$$= \Delta_f H_m^\Theta[\text{NdCl}_3 \cdot 6\text{H}_2\text{O(s)}] + 2\Delta_f H_m^\Theta[\text{Hhq(s)}]$$
$$+ \Delta_f H_m^\Theta[\text{NaAc(s)}] - \Delta_f H_m^\Theta[\text{NaCl(s)}]$$
$$- 2\Delta_f H_m^\Theta[\text{HCl(g)}] - 6\Delta_f H_m^\Theta[\text{H}_2\text{O(l)}] + \Delta_r H_m^\Theta(1)$$

Table 8
Ancillary data[d]

Compound	Enthalpies (kJ mol^{-1})	References
$\Delta_f H_m^\Theta$ [LaCl$_3$·7H$_2$O(s)]	−(3178.6 ± 0.1)	[12,13]
$\Delta_f H_m^\Theta$ [PrCl$_3$·6H$_2$O(s)]	−(2880.7 ± 0.1)	[12,14]
$\Delta_f H_m^\Theta$ [NdCl$_3$·6H$_2$O(s)]	−(2874.4 ± 0.1)	[12,15]
$\Delta_f H_m^\Theta$ [SmCl$_3$·6H$_2$O(s)]	−(2870.2 ± 0.1)	[12,15]
$\Delta_f H_m^\Theta$ [8-hydroxyquinoline (s)]	−(83.0 ± 1.5)	[16]
$\Delta_f H_m^\Theta$ [NaAc(s)]	−708.13	[17,18]
$\Delta_f H_m^\Theta$ [NaCl(s)]	−411.12	[17,18]
$\Delta_f H_m^\Theta$ [HCl(g)]	−(92.31 ± 0.13)	[19]
$\Delta_f H_m^\Theta$ [H$_2$O(l)]	−(285.830 ± 0.042)	[19]
$\Delta_s H_\infty^\Theta$ [HCl(g)]	74.772	[17]
$\Delta_d H_\infty^\Theta$ [HCl(aq, 4.000→0]	4.397	[17]

[d] $\Delta_f H_m^\Theta$ was the standard molar enthalpy of formation, $\Delta_s H_\infty^\Theta$ was the enthalpy of infinite dilution, and $\Delta_d H_\infty^\Theta$ was the enthalpy of dilution, respectively.

$$= [-2874.4] + 2[-83.0] + [-708.13] - [-411.12]$$
$$- 2[-92.31] - 6[-285.830] + [-56.9]$$
$$= -(1494.7 \pm 3.3)$$

$$\Delta_f H_m^\Theta[\text{Sm(hq)}_2\text{Ac(s)}]$$
$$= \Delta_f H_m^\Theta[\text{SmCl}_3 \cdot 6\text{H}_2\text{O(s)}]\Delta_f H_m^\Theta + 2[\text{Hhq(s)}]$$
$$+ \Delta_f H_m^\Theta[\text{NaAc(s)}] - \Delta_f H_m^\Theta[\text{NaCl(s)}]$$
$$- 2\Delta_f H_m^\Theta[\text{HCl(g)}] - 6\Delta_f H_m^\Theta[\text{H}_2\text{O(l)}] + \Delta_r H_m^\Theta(2)$$
$$= [-2870.2] + 2[-83.0] + [-708.13] - [-411.12]$$
$$- 2[-92.31] - 6[-285.830] + [-67.9]$$
$$= -(1501.5 \pm 3.4)$$

The uncertainties of $\Delta_f H_m^\Theta[\text{Ln(hq)}_2(\text{Ac})(\text{s})]$ can be calculated from the following equation:

$$s\,\Delta_f H_m^\Theta[\text{Ln(hq)}_2\text{Ac(s)}]$$
$$= [\{s\,\Delta_f H_m^\Theta[\text{LnCl}_3 \cdot 6\text{H}_2\text{O(s)}]\}^2 + \{2s\,\Delta_f H_m^\Theta[\text{Hhq(s)}]\}^2$$
$$+ \{s\,\Delta_f H_m^\Theta[\text{NaAc(s)}]\}^2 + \{s\,\Delta_f H_m^\Theta[\text{NaCl(s)}]\}^2$$
$$+ \{2s\,\Delta_f H_m^\Theta[\text{HCl(g)}]\}^2 + \{6s\,\Delta_f H_m^\Theta[\text{H}_2\text{O(l)}]\}^2$$
$$+ \{s\,\Delta_r H_m^\Theta\}^2]^{1/2}$$

where $s\,\Delta_f H_m^\Theta$ was the corresponding uncertainty of the standard molar enthalpy of formation, and $s\,\Delta_r H_m^\Theta$ was the corresponding uncertainty of the standard molar enthalpy of reaction.

Table 7
The standard molar enthalpies of reaction for the two reactions[c]

Reaction	$\Delta_r H_m^\Theta$ (kJ mol^{-1})
NdCl$_3$·6H$_2$O(s) + 2Hhq(s) + NaAc(s) = Nd(hq)$_2$Ac(s) + NaCl(s) + 2HCl(g) + 6H$_2$O(l)	−(56.9 ± 1.4)
SmCl$_3$·6H$_2$O(s) + 2Hhq(s) + NaAc(s) = Sm(hq)$_2$Ac(s) + NaCl(s) + 2HCl(g) + 6H$_2$O(l)	−(67.9 ± 1.5)

[c] $\Delta_r H_m^\Theta$ was the standard molar enthalpy of reaction, and the uncertainty quoted for $\Delta_r H_m^\Theta$ corresponded to the standard deviation.
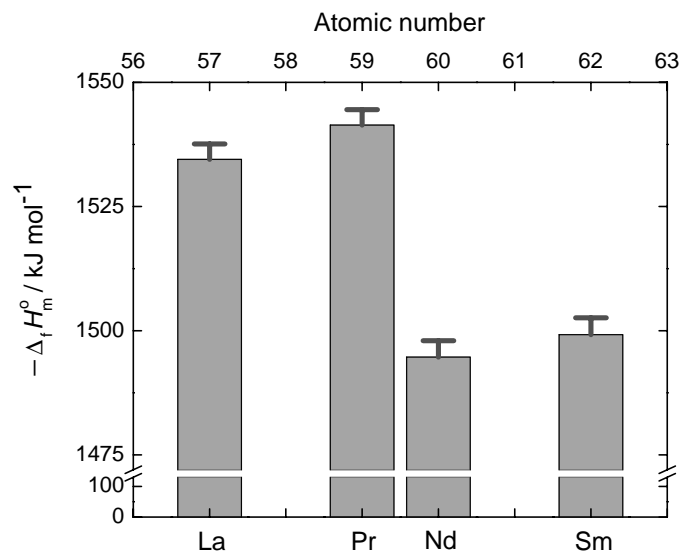
Atomic number

Fig. 2. The standard molar enthalpies of formation in dependence on the atomic number.

Table 9
The standard molar enthalpies of formation of of Ln(hq)$_2$Ac (Ln: La, Pr, Nd and Sm)

Compound	$\Delta_f H_m^{\ominus}$ (kJ mol^{-1})
La(hq)$_2$Ac(s)	$-(1534.5 \pm 3.1)$
Pr(hq)$_2$Ac(s)	$-(1541.4 \pm 3.1)$
Nd(hq)$_2$Ac(s)	$-(1494.7 \pm 3.3)$
Sm(hq)$_2$Ac(s)	$-(1501.5 \pm 3.4)$

The standard molar enthalpies of formation of La(hq)$_2$Ac(s) and Pr(hq)$_2$Ac(s), $\Delta_f H_m^{\ominus}$[La(hq)$_2$Ac(s)] and $\Delta_f H_m^{\ominus}$ [Pr(hq)$_2$Ac(s)], were determined in our previous study [7]. The values of them were recalculated by using the new values of the standard molar enthalpies of formation for LnCl$_3 \cdot n$H$_2$O(s) (Ln: La, Pr; for La, $n = 7$ and for Pr, $n = 6$), 8-hydroxyquinoline and NaCl. There are no reported associated uncertainties of $\Delta_f H_m^{\ominus}$ for NaAc and NaCl, so we did not consider the uncertainties of them in calculation of $\Delta_f H_m^{\ominus}$ of Ln(hq)$_2$Ac (Ln: La, Pr, Nd and Sm). The final results were summarized in Table 9 for comparison. The standard molar enthalpies of formation of Ln(hq)$_2$Ac (Ln: La, Pr, Nd and Sm) in dependence on the atomic number was plotted in Fig. 2. The little difference among the standard molar enthalpies of formation of Ln(hq)$_2$Ac (Ln: La, Pr, Nd and Sm) may be attributed to the lanthanide contraction.

4. Conclusion

In the present work, the standard molar enthalpies of formation of Nd(hq)$_2$Ac and Sm(hq)$_2$Ac, at $T = 298.15$ K, have been derived to be $-(1494.7 \pm 3.3)$ and $-(1501.5 \pm 3.4)$ kJ mol^{-1}, respectively. The standard molar enthalpies of formation of similar compounds were compared and discussed.

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