Determination of the standard molar enthalpy of formation of the ternary complex of neodymium with vitamin B3 and 8-hydroxylquinoline by microcalorimetry

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Abstract A novel solid ternary complex, [Nd(C$_6$H$_4$NO$_2$)$_2$·C$_9$H$_6$NO·2H$_2$O], was synthesized in a water bath (333.15 K) by three kinds of reagents: neodymium chloride, vitamin B3 (C$_6$H$_5$NO$_2$), and 8-hydroxylquinoline (C$_9$H$_7$NO). Its composition and structure were characterized by elemental analysis, IR spectra, UV spectra, molar conductance, and thermogravimetric analysis. During the process of coordination, C$_6$H$_5$NO$_2$ was bidentate-coordinated with Nd^{3+} in the form of an acidic group by removing the proton; hydroxyl oxygen atom and heterocyclic nitrogen atom of C$_9$H$_7$NO formed a chelate ring. Particularly, in this article, a thermochemical cycle in the calorimetric solvent (V_{HCl} : V_{DMF} : V_{EtOH} = 3:1:1) was designed on the basis of Hess's law. At 298.15 K, the dissolution enthalpies of the reactants and products were determined by a advanced solution–reaction isoperibol microcalorimeter, respectively. According to the above results and relevant literature data, the standard molar enthalpy of formation of [Nd(C$_6$H$_4$NO$_2$)$_2$·C$_9$H$_6$NO·2H$_2$O], was estimated to be $\Delta_f H_m^\Theta$ [[Nd(C$_6$H$_4$NO$_2$)$_2$·C$_9$H$_6$NO·2H$_2$O(s)], 298.15 K] = −(2,129.1 ± 2.5) kJ mol^{-1}.

Introduction

At present, the abuse of drugs, especially misuse of antibiotics, has led to the spread of resistant bacteria proliferation, which has been a topic of common concern. But the development of bacteria-resistant drugs cannot meet the demands of the public. Interestingly, recent reports have showed that rare earth ions (RE^{3+}) have unique physiological and biochemical characteristics. And they possess the functions of antisepsis, anti-inflammation, and anticancer [1, 2]. Due to their strong affinities to many biological molecules, rare earth ions can effectively participate in many important life processes and activate or inhibit a variety of enzymes or proenzymes. Vitamin B3 (C$_6$H$_5$NO$_2$), known as vitamin PP, one of the body's thirteen essential vitamins, can participate in some vital processes in human body, such as lipid metabolism, the oxidation of tissue respiration, and breakdown of glycogen [3]. In addition, complexing ligands such as 8-hydroxylquinoline and their derivatives were found to have potential bioactivities including anticancer, antibacterial, and antioxidative properties, vasorelaxing properties, antivirus, and antiplatelet activities [4]. Thus, we could conjecture that the combination of the three forementioned substances may represent stronger bioactivities than those of the rare earth, vitamin B3, and 8-hydroxyquinoline alone [5, 6].

A large number of experimental results indicated that compared with organic synthetic drugs and transition metal coordination compounds, the products from the reactions of rare earth with many organic compounds have strong

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effects of sterilization and bacteriostasis, importantly of being lower toxicity and less accumulation in the body. Given the advantages above, a lot of researches in this field have been done recently, but the study of thermodynamic rare earth complexes is still not perfect. Therefore, in this article, the synthesis and characterization of a new ternary complex of neodymium chloride with vitamin B3 and 8-hydroxyquinoline were reported and its thermodynamic properties were investigated. This type of study, we believe, may potentially benefit for the research and development of rare earth coordination. In the aspect of medicinal study to screen the rare earth complexes of high-efficiency and low toxicity, it also has an important significance theoretically and practically.

Experimental

Reagents and instruments

Reagents

$NdCl_3 \cdot 6H_2O$ (>99.5 %, produced by Chengdu Fei Tian Co., Ltd., recrystallized with triple distilled water); Vitamin B3 (>99.5 %, A.R., produced by the Shanghai Reagent Company, recrystallized with anhydrous ethanol); and C_9H_7NO (>99.5 %, A.R., obtained from Tianjin Guangfu Fine Chemical Research Institute, recrystallized with anhydrous ethanol); These samples were dried in a vacuum desiccator containing P_4O_{10} to constant mass. HCl (A.R.); NaOH (A.R.); dimethylformamide (DMF, A.R.); ethanol (EtOH, A.R.).

Instruments

Elemental analyzer (Perkin-Elmer 2400 CHN, USA); thermogravimetry analysis instrument (STA449C, NET-ZSCH Corporation, Germany); Fourier IR spectrometer (Avatar360, Nicolet, USA, with KBr pellet); UV–Visible spectrophotometer (U-3010, HITACHI, Japan); digital Abbe refractometer (WAY-IS, China); the dissolution enthalpies were measured by an isoperibol calorimeter (SRC-100, constructed by the Thermochemical Laboratory of Wuhan University, China) [7], the precisions of the control temperature and test temperature are ±0.001 and ±0.0001 K, respectively. The calibration of the calorimeter was carried out by measuring the dissolution enthalpies of KCl (calorimetric primary standard) in triple distilled water and trihydroxymethyl aminomethane (THAM, NBS 742a, USA) in 0.0001 mol mL^{-1} HCl at 298.15 K. This meant that dissolution enthalpies were (17,597 ± 17) J mol^{-1} for KCl and −(29,776 ± 16) J mol^{-1} for THAM, which agreed with published data [(17,536 ± 9) J mol^{-1} for KCl and −(29,766 ± 31.5) J mol^{-1} for THAM] [7, 8]. The

eventual error was less than 0.5 %, which suggested the calorimeter was reliable.

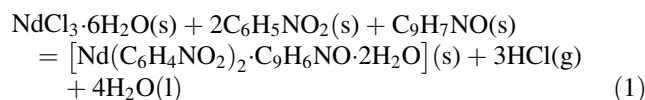### Synthesis of complex

A certain amount of NaOH was added into $C_6H_5NO_2$/ethanol solution under the condition of slight heating. Then the sodium salt solution (formed in the former procedure) and C_9H_7NO/ethanol solution were mixed. At 333.15 K, neodymium chloride/ethanol solution was added dropwise to the mixture during being stirred, the final solution was settled down at pH 6.5–7.0. And then, the reaction solution was stirred for 6 h. After overnight deposition, air pump filtration and washed alternatively with ethanol and water at 353.15 K, until it was not detected Cl$^-$ existing in the filtrate, the solid complex was obtained. More detailed description was available in the Ref. [9]. The product was dried at 333.15 K for 24 h and kept until the mass of it became a constant. The chemical composition of product was measured by elemental analysis for C, H, and N, by EDTA titration for Nd^{3+} ion, by TG–DTG for coordinated water molecules. The results showed that the formula of complex was [Nd($C_6H_4NO_2$)$_2 \cdot C_9H_6NO \cdot 2H_2O$].

Thermochemical cycle of the coordination reaction

Because of having difficult in determining the thermal effect of solid state coordination reaction, it was feasible to derive the enthalpies of formation by measuring the dissolution enthalpies when the samples were dissolved. The thermochemical studies were carried out by an isoperibol calorimeter (SRC-100, constructed by the Thermochemical Laboratory of Wuhan University, China). In the process, at 298.15 K, the current was 11.760 mA, and the resistance of heater was 1,251.5 Ω. Through Hess's law, a convincing thermochemical cycle was designed and showed in Fig. 1.

In Fig. 1, the equation of the coordination reaction is as follows:

$$NdCl_3 \cdot 6H_2O(s) + 2C_6H_5NO_2(s) + C_9H_7NO(s)$$
$$= \left[Nd(C_6H_4NO_2)_2 \cdot C_9H_6NO \cdot 2H_2O\right](s) + 3HCl(g)$$
$$+ 4H_2O(l) \tag{1}$$

Then the spectrum and refractive indexes the solution C and the solution F were determined, finding that both were the similar UV spectra curves and equal refractive indexes, which demonstrated that they had the same thermodynamics state. And the thermochemical cycle of the coordination reaction designed was reliable.

Selection of calorimetric solvent

It was very important to choose a solvent, which should dissolve chemicals rapidly and completely. By texting at

Fig. 1 Thermochemical cycle of the coordination reaction
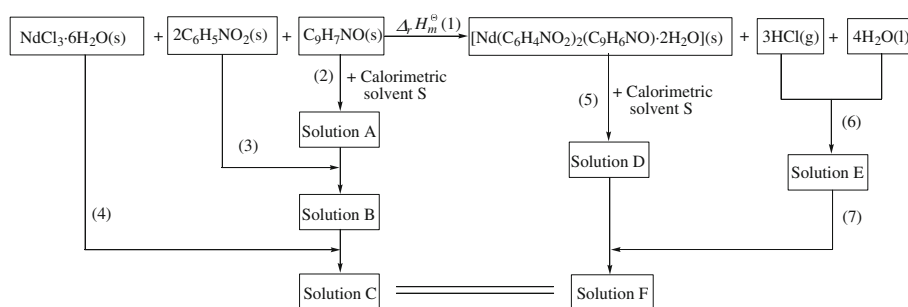
different ratio, the calorimetric solvent S ($V_{HCl}:V_{DMF}:V_{EtOH} = 3:1:1$) was the most appropriate solvent for this experiment.

Determine of dissolution enthalpies of reactants and products

The thoroughly dried samples were ground well in an agate mortar, and a sample (0.25 mmol) was placed into the sample container of calorimeter. The calorimetric solvent S (100.00 mL) was added into the reaction vessel in advance. When the calorimeter was adjusted to a constant temperature of (298.150 ± 0.001) K, the dissolution enthalpies were measured. The results were showed in Table 1, after five parallel measurements. More detailed description was available in the Ref. [10].

Results and discussion

UV spectra of complex

The complex, the free ligands $C_6H_5NO_2$ and C_9H_7NO in the mixed solvent ($V_{DMSO}:V_{EtOH} = 1:9$) were measured by UV spectra, respectively. As shown in Fig. 2, ligand $C_6H_5NO_2$ had the maximum absorption at the 263 nm, which was the $\pi–\pi^*$ transition of the carboxyl and aromatic heterocyclic. Free ligand C_9H_7NO had a sharp and strong absorption peak at 246 nm due to the condensed nucleus $\pi–\pi^*$ transition absorption. Additionally, there was a weaker broad peak at 316 nm because of the absorption
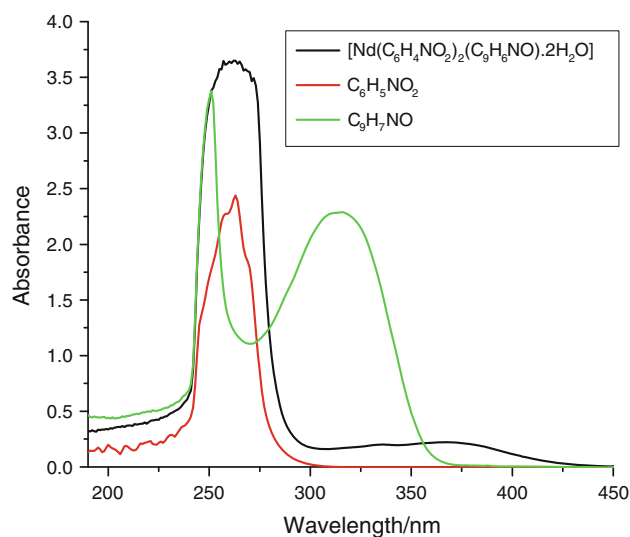
Fig. 2 The UV of [Nd(C$_6$H$_4$NO$_2$)$_2$(C$_9$H$_6$NO)·2H$_2$O], C$_6$H$_5$NO$_2$, and C$_9$H$_7$NO

spectrum of the $n–\pi^*$ transition of the phenolic hydroxyl oxygen and cyclobenzene.

The UV spectra of synthetic complex was obviously different from free ligands. It can be found that the synthetic complex had a strong absorption band ($\varepsilon > 3,500$ L mol^{-1} cm^{-1}) at 240–290 nm, which was stronger and broader than that of every free ligand and it totally covered the absorption bands of the two free ligands. In addition, with increase of the number of the aromatic rings of complex and the conjugate degree of the π bond, the bigger delocalization conjugate system would form, which

Table 1 Dissolution enthalpies of [2C$_6$H$_5$NO$_2$(s)], [C$_9$H$_7$NO(s)], [NdCl$_3$·6H$_2$O(s)], [Nd(C$_6$H$_4$NO$_2$)$_2$·C$_9$H$_6$NO·2H$_2$O(s)], [solution E (aq)] in the calorimetric solvent S at 298.15 K $\Delta_s H_m^{\ominus}$ (kJ mol^{-1})

Sample	2C$_6$H$_5$NO$_2$(s)	C$_9$H$_7$NO(s)	NdCl$_3$·6H$_2$O(s)	[Nd(C$_6$H$_4$NO$_2$)$_2$·C$_9$H$_6$NO·2H$_2$O](s)	Solution E
1	35.579	−3.503	−34.819	−49.414	−10.554
2	35.466	−3.967	−34.433	−50.312	−10.266
3	35.549	−3.728	−34.456	−49.626	−11.028
4	36.254	−3.235	−34.034	−49.429	−10.699
5	35.154	−3.742	−34.401	−48.803	−10.704
Avg.	35.600 ± 0.403	−3.635 ± 0.277	−34.429 ± 0.278	−49.517 ± 0.541	−10.650 ± 0.276

led to red shift and had stronger absorption band of the $\pi-\pi^*$ transition of the complex. From the view of quantum mechanics, if the value of ε was bigger than 10^4 L mol^{-1} cm^{-1}, transition can absolutely occur. When it ranged from 10^3 to 10^4, the probability of the $\pi-\pi^*$ transition of the complex is great. The complex had a weak absorption band in the 300–430 nm, so the complex showed light yellow.

IR spectra of complex

The IR spectra of $C_6H_5NO_2$, C_9H_7NO, and the complex was determined by FT-IR spectrometer with KBr pellet between 4,000 and 400 cm^{-1}, and shown in Fig. 3.

There were three characteristic absorption bands observed for free $C_6H_5NO_2$: ν_{O-H} (2,200–2,700 cm^{-1}), $\nu_{C=O}$ (1,715 cm^{-1}), and δ_{oop} (O–H 952 cm^{-1}). After formation the complex, three characteristic absorption bands of carboxylate groups vanished. At the same time, due to the carboxylate group (COO$^-$), two new absorption bands: ν_{as} (1,597 cm^{-1}) and ν_s (1,403 cm^{-1}), appeared. The results proved that carboxylic acid was bidentate-coordinated with Nd^{3+} in the form of acidic group after being removed the proton of carboxylic acid.

There were three characteristic bands for C_9H_7NO: ν_{O-H} (3,136 cm^{-1}), δ_{ip} (O–H 1,379 cm^{-1}, S), $\nu_{C=N+C=C}$ (1,576, 1,505, and 1,471 cm^{-1}). The ν_{O-H} and δ_{O-H} vanished after the complex formed, which certified that C_9H_7NO was coordinated with Nd^{3+} after remove of the hydrogen of hydroxyl group. Besides, the skeleton vibration peak of quinoline ring shifted to low wave number with 6 cm^{-1}, indicating that the hydroxyl oxygen atom and heterocyclic

nitrogen atom of $C_9H_6NO^-$ were bidentate-coordinated with Nd^{3+} and formed a five-membered chelate ring, which increased the conjugation degree of quinoline ring, yet decreased the bond strength of C=N and C=C.

The single IR absorption band at 422 cm^{-1} was assigned to the stretching vibration of the Nd–O bond. The characteristic absorption band of ν_{O-H} of H_2O appeared between 3,550 and 3,000 cm^{-1}. And H–OH bending vibration band at 1,625 cm^{-1} further illustrated the existence of water in the complex. According to the data from elemental analysis and TG–DTG, there were two water molecules coordinated to Nd^{3+} in the complex.

Mechanism of thermal decomposition

TG–DTG curves ($\beta = 20$ °C min^{-1}, temperature ranging from room temperature to 935 °C, N_2 atmosphere) of [Nd($C_6H_4NO_2$)$_2$·C_9H_6NO·$2H_2O$] were shown in Fig. 4. The thermal decomposition process could be divided into three steps. The first decomposition process started from 137.1 to 180.5 °C with a mass loss of 6.41 % corresponding to the loss of 2 mol H_2O (theoretical loss: 6.33 %). The second decomposition process started from 197.6 to 611.0 °C with a mass loss of 41.54 % corresponding to the loss of 2 mol $C_6H_4NO_2^-$ (theoretical loss 40.14 %). The characteristic absorption band of ν_{as}(COO$^-$) disappeared by measuring the IR spectra of the residue, indicating the $C_6H_4NO_2^-$ of the complex was completely lost. The third decomposition process lost a part mass of $C_9H_6NO^-$ with 9.43 %. So the complex did not be decomposed entirely in the 935 °C. The salicylic acid is melted at 236 °C. 8-Hydroxyquinoline is melted at 72 °C
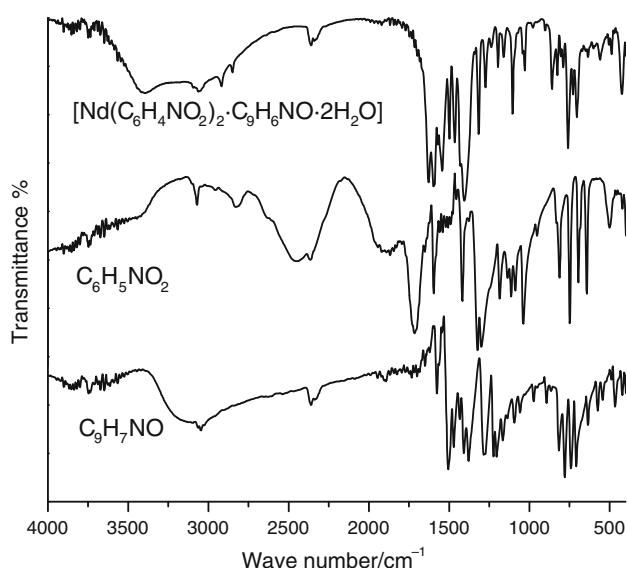

Fig. 3 The IR spectra of the free ligands and the synthetic complex
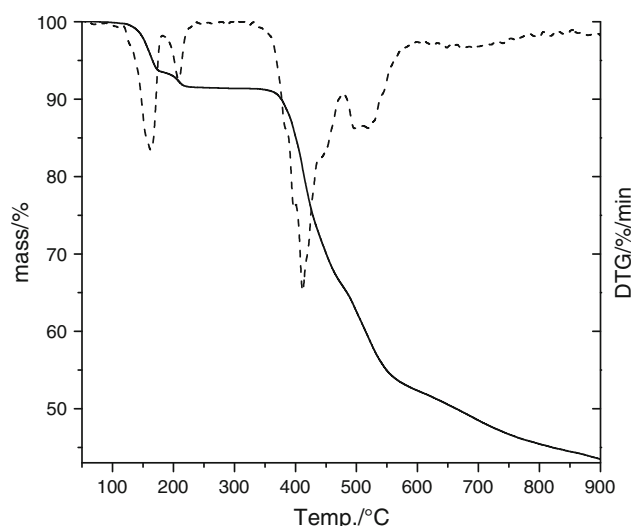
Fig. 4 TG–DTG curves ($\beta = 20$ °C min^{-1}) of the complex [Nd($C_6H_4NO_2$)$_2$·C_9H_6NO·$2H_2O$]
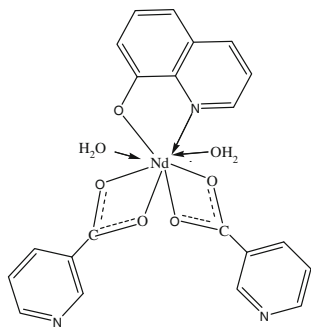
Fig. 5 Structure schematic diagram of the complex $[Nd(C_6H_4NO_2)_2\cdot C_9H_6NO\cdot 2H_2O]$

and is gasified to decompose at 207 °C. The higher decomposition temperature suggested that the complex had a better thermal stabilization.

Elemental analysis and putative structure of the complex

By the elemental analysis, found the theoretical values of the elemental analysis for $[Nd(C_6H_4NO_2)_2\cdot C_9H_6NO\cdot 2H_2O]$ are the following: C, 44.36 %; H, 3.19 %; N, 7.39 %; Nd, 25.37 %. Found values are the following: C, 44.20 %; H, 3.28 %; N, 7.24 %; Nd, 25.65 %.

The molar conductance was determined to be 13.2 S cm^2 mol^{-1}, proving that the complex existed in the form of nonelectrolyte in DMSO. The complex was light yellow powder and very stable in the atmosphere. It can be dissolved in dimethyl sulfoxide and *N,N*-dimethylformamide but cannot be dissolved in H_2O, chloroform, methanol, ethanol, acetone, petroleum ether, or tetrahydrofuran.

As above mentioned, the putative structure of the complex was shown in Fig. 5.

Data treatment of calorimetric experiment

Evaluation of $\Delta_s H_m^{\ominus}(6)$

According to reaction (6)

$$3HCl(g) + 4H_2O(l) \rightarrow solution\,E$$
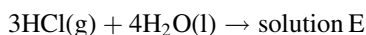
The molality of solution E is 41.667 mol kg^{-1}.
According to Ref. [11],

$$\Delta_s H_m^{\ominus}(6) = -39.920\,kJ\,mol^{-1}.$$

The standard molar reaction enthalpy of the coordination reaction

According to Hess's law, the standard molar reaction enthalpy of the coordination reaction was obtained

$$\Delta_r H_m^{\ominus}(1) = \Delta_s H_m^{\ominus}(2) + \Delta_s H_m^{\ominus}(3) + \Delta_s H_m^{\ominus}(4) - \Delta_s H_m^{\ominus}(5)$$
$$- \Delta_s H_m^{\ominus}(6) - \Delta_s H_m^{\ominus}(7) = 35.600 + (-34.429)$$
$$+ (-3.635) - (-49.517) - (-39.920) - (-10.650)$$
$$\pm \left[\sqrt{0.403^2 + 0.278^2 + 0.277^2 + 0.541^2 + 0.276^2} \right]$$
$$= (97.62 \pm 0.83)\,kJ\,mol^{-1}.$$

The standard molar enthalpies of formation of the complex

According to Hess's law and principles of thermodynamics

$$\Delta_r H_m^{\ominus}(1)$$
$$= \Delta_f H_m^{\ominus}[Nd(C_6H_4NO_2)_2\cdot C_9H_6NO\cdot 2H_2O(s), 298.15\,K]$$
$$+ 3\Delta_f H_m^{\ominus}[HCl(g), 298.15\,K] + 4\Delta_f H_m^{\ominus}[H_2O(l), 298.15\,K]$$
$$- 2\Delta_f H_m^{\ominus}[C_6H_5NO_2(s), 298.15\,K]$$
$$- \Delta_f H_m^{\ominus}[C_9H_7NO(s), 298.15\,K]$$
$$- \Delta_f H_m^{\ominus}[NdCl_3\cdot 6H_2O(s), 298.15\,K.]$$

According to Refs. [12–15],

$$\Delta_f H_m^{\ominus}[HCl(g), 298.15\,K] = -(92.31 \pm 0.10)\,kJ\,mol^{-1}$$
$$\Delta_f H_m^{\ominus}[H_2O(l), 298.15\,K] = -(285.83 \pm 0.04)\,kJ\,mol^{-1}$$
$$\Delta_f H_m^{\ominus}[C_6H_5NO_2(s), 298.15\,K] = -(344.81 \pm 0.92)\,kJ\,mol^{-1}$$
$$\Delta_f H_m^{\ominus}[C_9H_7NO(s), 298.15\,K] = -(83.0 \pm 1.5)\,kJ\,mol^{-1}$$
$$\Delta_f H_m^{\ominus}[NdCl_3\cdot 6H_2O(s), 298.15\,K] = -2,874.4\,kJ\,mol^{-1}$$

Therefore,

$$\Delta_f H_m^{\ominus}[Nd(C_6H_4NO_2)_2\cdot C_9H_6NO\cdot 2H_2O](s), 298.15\,K]$$
$$= 97.62 - 3 \times (-92.31) - 4 \times (-285.83) + 2$$
$$\times (-344.81) + (-83.0) + (-2,874.4)$$
$$\pm \sqrt{0.83^2 + (3 \times 0.1)^2 + (4 \times 0.04)^2 + (2 \times 0.92)^2 + 1.5^2}$$
$$= -(2,129.1 \pm 2.5)\,kJ\,mol^{-1}.$$

Experimental discussions

The precision of the control temperature and test temperature were ±0.001 and ±0.0001 K, respectively. In the calculation of standard molar reaction enthalpy of the coordination reaction, it was inevitable for small amount of HCl volatilization, but it had little effect on the experiment by calorimetry, which could be neglected.

Conclusions

The standard molar enthalpy of formation of the ternary complex of neodymium with vitamin B3 and 8-hydroxylquinoline was determined by microcalorimetry. The

composition of the complex was $[Nd(C_6H_4NO_2)_2 \cdot C_9H_6NO \cdot 2H_2O]$ and its purity was more than 99.0 %, which were proved by elemental analysis, UV spectra, and IR spectra. Furthermore, according to Hess's law, a thermochemical cycle was designed. The dissolution enthalpies of $[NdCl_3 \cdot 6H_2O(s)]$, $[2C_6H_5NO_2(s)]$, $[C_9H_7NO(s)]$, $[Nd(C_6H_4NO_2)_2 \cdot C_9H_6NO \cdot 2H_2O(s)]$, were derived by a solution–reaction isoperibol microcalorimeter. The results showed that $\Delta_s H_m^\Theta [NdCl_3 \cdot 6H_2O(s), 298.15 \ K] = -(34.429 \pm 0.278) \ kJ \ mol^{-1}$, $\Delta_s H_m^\Theta [2C_6H_5NO_2(s), 298.15 \ K] = (35.600 \pm 0.403) \ kJ \ mol^{-1}$, $\Delta_s H_m^\Theta [C_9H_7NO(s), 298.15 \ K] = -(3.635 \pm 0.277) \ kJ \ mol^{-1}$, and $\Delta_s H_m^\Theta [Nd(C_6H_4NO_2)_2 \cdot C_9H_6NO \cdot 2H_2O(s), 298.15 \ K] = -(49.517 \pm 0.541) \ kJ \ mol^{-1}$. The standard molar enthalpy change of the reaction was determined based on the experimental data to be $\Delta_r H_m^\Theta(1) = (97.62 \pm 0.83) \ kJ \ mol^{-1}$, and the standard molar enthalpy of formation of the synthetic coordination compound was calculated to be $\Delta_f H_m^\Theta [[Nd(C_6H_4NO_2)_2 \cdot C_9H_6NO \cdot 2H_2O](s), 298.15 \ K] = -(2,129.1 \pm 2.5) \ kJ \ mol^{-1}$. Meanwhile, in this study, we gave structural schematic diagram of the complex $[Nd(C_6H_4NO_2)_2 \cdot C_9H_6NO \cdot 2H_2O]$, which could help us to understand the coordination process of the complex.

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