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Low-temperature heat capacities and standard molar enthalpy of formation of the solid-state coordination compound *trans*-Cu(Ala)$_2$(s) (Ala = L-α-alanine)

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ABSTRACT

Low-temperature heat capacities of the solid coordination compound *trans*-Cu(Ala)$_2$(s) have been measured by a precision automated adiabatic calorimeter over the temperature range from $T = 78$ K to 390 K. The experimental values of the molar heat capacities in the temperature region were fitted to a polynomial equation of heat capacities ($C_{p,\mathrm{m}}$) with the reduced temperatures (X), [$X = f(T)$], by a least square method. The smoothed molar heat capacities and thermodynamic functions of the complex *trans*-Cu(Ala)$_2$(s) were calculated based on the fitted polynomial. The smoothed values of the molar heat capacities and fundamental thermodynamic functions of the sample relative to the standard reference temperature 298.15 K were tabulated with an interval of 5 K. Enthalpies of dissolution of $\{\mathrm{Cu(Ac)_2{\cdot}H_2O(s)+2Ala\ (s)}\}$ and 2:1 HAc (aq) in 100 ml of 2 mol dm^{-3} HCl, respectively, and *trans*-Cu(Ala)$_2$(s) in the solvent [2:1 HAc (aq) + 2 mol dm^{-3} HCl] at $T = 298.15$ K were determined to be $\Delta_\mathrm{S}H^\circ_\mathrm{m}[\mathrm{Cu(Ac)_2 \cdot H_2O(s) + 2Ala(s)}] = (6.75 \pm 0.04)$ kJ mol^{-1}, $\Delta_\mathrm{S}H^\circ_\mathrm{m}[2:1\ \mathrm{HAc(aq)}] = -(1.63 \pm 0.01)$ kJ mol^{-1}, and $\Delta_\mathrm{S}H^\circ_\mathrm{m}[\textit{trans}\text{-}\mathrm{Cu(Ala)_2(s)}] = -(10.24 \pm 0.08)$ kJ mol^{-1} by means of an isoperibol solution-reaction calorimeter. The standard molar enthalpy of formation of the compound was determined as $\Delta_\mathrm{f}H^\circ_\mathrm{m}(\textit{trans}\text{-}\mathrm{Cu(Ala)_2(s)}, 298.15\ \mathrm{K}) = -(1038.6 \pm 3.5)$ kJ mol^{-1} from the enthalpies of dissolution and other auxiliary thermodynamic data using a Hess thermochemical cycle.

1. Introduction

Solid-state chemical synthesis is an important and promising method in the field of synthetic chemistry. The method [1] has been widely used to synthesize various solid-state coordination compounds of many metal salts with α-amino acids because it avoids complicated experimental operations and harsh reaction conditions. It is well-known that α-amino acids are basic structural units which constitute proteins, and copper(II) is one of the trace elements necessary for the human body. It was established early that the complexes of α-amino acids with copper (II) formed some compounds with *cis*- and *trans*-geometric isomers, which have extensive application in food, cosmetics and medicines as a nutrient.

In 1993, Zheng and Xin [2] synthesized a series of complexes (including *trans*-Cu(Ala)$_2$(s) and others) by means of solid-state coordination reactions of copper(II) acetate with α-amino acids at ambient temperature. XRD, IR, DTA and elemental analysis were employed to characterize their structures. However, up to now, the enthalpies of the solid-state coordination reactions involved in the syntheses of these complexes and their respective low-temperature heat capacities, thermodynamic functions and standard molar enthalpies of formation have not been reported in literature. The purpose of the present study is to determine the dissolution enthalpies of the reactants and the products of the solid-state coordination reaction of copper(II) acetate with L-α-alanine by isoperibol solution calorimetry, and measure the low-temperature heat capacities by adiabatic calorimetry. In addition, the thermodynamic functions and standard molar enthalpy of formation of the product of the solid-state coordination reaction [*trans*-Cu(Ala)$_2$(s)] were derived from these experimental results.

2. Experimental

2.1. Chemicals

Copper(II) acetate [Cu(Ac)$_2$·H$_2$O(s)], L-α-alanine(s) and other reagents used in these experiments were of analytical grade and

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obtained from Shanghai Reagent Factory. KCl(s) and α-Al_2O_3(s) used for calibration of the isoperibol solution calorimeter and adiabatic calorimeter were of a purity greater than 99.99%, and dried in a vacuum oven for 6 h at 210–240 °C prior to use. The water used in measurements was twice distilled.

2.2. Sample preparations and characterization

The samples used in the present measurements were prepared according to the method given in the literature [1,2], in which 10 mmol $Cu(Ac)_2 \cdot H_2O$(s) of 80–100 mesh was mixed and ground with a certain amount of L-α-alanine in a 1:2 mole ratio. These were retained for 4 h at ambient temperature. If subsequent XRD measurement showed that the refraction peaks of the two reactants in the system of the solid-state coordination reaction had disappeared, the reaction was complete. The product was washed three times with absolute ethyl alcohol, and once with ethyl ether of analytical grade. Finally, the sample was placed in a vacuum desiccator at ambient temperature to vacuum dry for 4 h. TG/DTG, chemical and elemental analyses (model: PE-2400, PerkinElmer, USA) have shown that the purity of the sample prepared was higher than 99.90 mol%.

2.3. Adiabatic calorimetry

A precision automatic adiabatic calorimeter was used to measure heat capacities over the temperature range $78 \le (T/\text{K}) \le 390$. The calorimeter was established in the Thermochemistry Laboratory of the Dalian Institute of Chemical Physics, Chinese Academy of Sciences, China. The principle and structure of the adiabatic calorimeter were described in detail elsewhere [3,4]. Briefly, the calorimeter mainly comprised a sample cell, a platinum resistance thermometer, an electric heater, inner and outer adiabatic shields, two sets of six-junction chromel-constant an thermopiles installed between the calorimetric cell and the inner shield and between the inner and outer shields, respectively, and a high vacuum can. The miniature platinum resistance thermometer (IPRT No. 2, produced by Shanghai Institute of Industrial Automatic Meters, 16 mm in length, 1.6 mm in diameter and a nominal resistance of 100 Ω) was applied to measure the temperature of the sample. The thermometer was calibrated on the basis of ITS-90 by the Station of Low-Temperature Metrology and Measurements, Academia Sinica. The electrical energy introduced into the sample cell and the equilibrium temperature of the cell after the energy input were automatically recorded by use of a Data Acquisition/Switch Unit (Model 34970A, Agilent, USA), and processed on line by a computer.

To verify the accuracy of the calorimeter, the heat capacities of the reference standard material (α-Al_2O_3) were measured over the temperature range $78 \le (T/\text{K}) \le 390$. The sample mass used was 1.7143 g, which was equivalent to 0.0168 mol based on its molar mass, $M(Al_2O_3) = 101.9613\ \text{g mol}^{-1}$. Deviations of the experimental results from those of the smoothed curve lie within ±0.2%, while the uncertainty is ±0.3%, as compared with the values given by the former National Bureau of Standards [5] over the whole temperature range.

Heat-capacity measurements were continuously and automatically carried out by means of the standard method of intermittently heating the sample and alternately measuring the temperature. The heating rate and temperature increments were generally controlled at $(0.1–0.4)\ \text{K min}^{-1}$ and (1–3) K. The heating duration was 10 min, and the temperature drift rates of the sample cell measured in an equilibrium period were always kept within $(10^{-3}$ to $10^{-4})\ \text{K min}^{-1}$ during the acquisition of all heat-capacity data. The data of heat capacities and corresponding equilibrium temperature have been corrected for heat exchange of the sample cell with its surroundings [3,4]. The sample mass used for calorimetric measurements was 3.3156 g, which was equivalent to 0.0137 mol in terms of its molar mass, $M = 241.73\ \text{g mol}^{-1}$.

2.4. Isoperibol solution-reaction calorimetry

The isoperibol solution-reaction calorimeter consisted primarily of a precision temperature controlling system, an electric energy calibration system, a calorimetric body, an electric stirring system, a thermostatic bath made from transparent silicate glass, a precision temperature measuring system and a data acquisition system. The principle and structure of the calorimeter were described in detail elsewhere [6].

The reliability of the calorimeter was verified by measuring the dissolution enthalpy of KCl (calorimetrically primary standard) in double distilled water at $T = 298.15$ K. The mean dissolution enthalpy was $\Delta_S H_m^\circ[\text{KCl(s)}] = (17,553 \pm 19)\ \text{J mol}^{-1}$ for KCl, as

Table 1

Dissolution enthalpies of $[Cu(Ac)_2 \cdot H_2O(s) + 2Ala(s)]$ and [2:1 HAc(aq)] in 2 mol dm^{-3} HCl and [*trans*-$Cu(Ala)_2$ (s)] in [2:1 HAc (aq) + 2 mol dm^{-3} HCl] at 298.15 K

System	Solvent	No.	m (g)	ΔE_e (mV)	ΔE_s (mV)	Q_e (J)	Q_s (J)	$\Delta_s H_m^o$ (kJ mol^{-1})
$Cu(Ac)_2 \cdot H_2O$ (s) + 2Ala (s)	2 mol L^{-1} HCl	1	0.377 5	1.25	1.60	5.28	6.76	6.76
		2	0.377 1	1.15	1.57	4.84	6.61	6.62
		3	0.377 9	1.14	1.61	4.85	6.84	6.86
		4	0.377 2	1.16	1.57	4.96	6.71	6.72
		5	0.377 2	1.15	1.60	4.86	6.77	6.78
$\Delta_s H_m^\circ[Cu(Ac)_2 \cdot H_2O(s) + 2Ala(s)] = (6.75 \pm 0.04)\text{kJ mol}^{-1}$								
2:1 HAc (aq)	2 mol L^{-1} HCl	1	0.122 1	1.47	−0.37	6.66	−1.68	−1.67
		2	0.122 7	1.42	−0.35	6.65	−1.64	−1.63
		3	0.122 3	1.42	−0.34	6.80	−1.63	−1.63
		4	0.122 5	1.41	−0.34	6.69	−1.61	−1.61
		5	0.122 0	1.40	−0.33	6.78	−1.60	−1.60
$\Delta_s H_m^\circ[\text{HAc(aq)}] = -(1.63 \pm 0.01)\text{kJ mol}^{-1}$								
Trans-$Cu(Ala)_2$ (s)	2:1 HAc (aq) + 2 mol dm^{-3} HCl	1	0.241 6	2.49	2.39	10.62	-10.19	-10.20
		2	0.241 0	2.50	2.35	10.62	- 9.98	-10.01
		3	0.241 8	2.51	2.41	10.63	-10.20	-10.20
		4	0.241 2	2.42	2.35	10.62	-10.31	-10.33
		5	0.241 5	2.40	2.37	10.61	-10.48	-10.48
$\Delta_s H_m^\circ \textit{trans}\text{-}Cu(Ala)_2(s) = -(10.24 \pm 0.08)\ \text{kJ mol}^{-1}$								

$R = 1666.0\ \Omega$, $I = 10.000$ mA, in which m (g) is mass of sample; ΔE_e (mV), the voltage change during the electrical calibration; ΔE_s (mV), the voltage change during the sample dissolution, Q_e (J), electrical energy of electrical calibration; Q_s (J), heat effect of the dissolution; $\Delta_s H_m^\circ = (\Delta E_s/\Delta E_e) I^2 R t_e (M/m)$, where R is the electro-resistance; I, the electrical current; M, the molar mass; t_e, heating period of electrical calibration.

shown in Table 1 (see supplementary data), which compared with corresponding published data [7] (17,536 ± 3.4) J mol^{-1}.

In all dissolution experiments of the sample, 100 ml of 2 mol dm^{-3} HCl was chosen as the calorimetric solvent for measuring the dissolution enthalpies of the $\{Cu(Ac)_2{\cdot}H_2O(s) + 2Ala(s)\}$ and the 2:1 HAc(aq) (HAc denotes the acetic acid), respectively. But the *trans*-$Cu(Ala)_2(s)$ was dissolved in the solvent [2:1 HAc (aq) + 2 mol dm^{-3} HCl] at $T = 298.15$ K.

The solid $Cu(Ac)_2{\cdot}H_2O(s)$ (Ac denotes the acetate) and Ala(s) were respectively ground within an agate mortar into a fine powder. The mixture of about 2 mmol Ala(s) and 1 mmol $Cu(Ac)_2{\cdot}H_2O(s)$ at mole ratio of $n(Cu(Ac)_2{\cdot}H_2O(s)){:}n(Ala) = 1{:}2$ was dissolved in 100 ml of 2 mol dm^{-3} HCl at $T = (298.15 \pm 0.001)$ K. The final solution obtained from five tests was designated as solution A.

About 0.12 g acetic acid solution, $n(HAc){:}n(H_2O) = 2{:}1$, was dissolved in 100 ml of 2 mol dm^{-3} HCl at $T = (298.15 \pm 0.001)$ K. The final solution was named as solution A_1.

The solid complex *trans*-$Cu(Ala)_2(s)$ was dried in a vacuum desiccator in order to take off some additional adsorbing water. Then, it was ground into a fine powder. The dissolution enthalpy of about 0.24 g of *trans*-$Cu(Ala)_2(s)$ in about 100 ml of the solution A_1 was determined under the same condition as the above. The final solution obtained was named as solution A'.

Finally, UV–vis spectroscopy and the data of the refractive indexes were used to confirm whether solution A was in the same thermodynamic state as that of solution A'. These results have indicated that the chemical components and physico-chemical properties of solution A were consistent with those of solution A'.

3. Results and discussion

3.1. Low-temperature heat capacities

All experimental results, listed in Table 2 (see supplementary data) and plotted in Fig. 1, showed that the structure of the coordination compound was stable over the temperature range between $T = 78$ K and 392 K, that is, no phase change, association nor thermal decomposition occurred. The 106 experimental points in the temperature region between $T = 78$ K and 392 K were fitted by means of the least squares method and a polynomial equation of the experimental molar heat capacities ($C_{p,m}$) vs. reduced temperature (X), $X = f(T)$, has been obtained,

$$C_{p,m}/\mathrm{J\,K^{-1}\,mol^{-1}} = 293.86 + 203.44X + 9.31X^2 - 10.72X^3 - 7.53X^4$$

In which $X = (T - 235)/157$. The standard deviations of experimental molar heat capacities from the smoothed heat capacities calculated by the polynomial equation were within ±0.3% except for several points around the lower and upper temperature limits. The correlation coefficient for the fitting R^2 equals 0.99994. The uncertainties of the coefficients of the equation have been determined to be 0.09%, 0.6%, 0.75%, 0.96% and 1.5%, respectively.

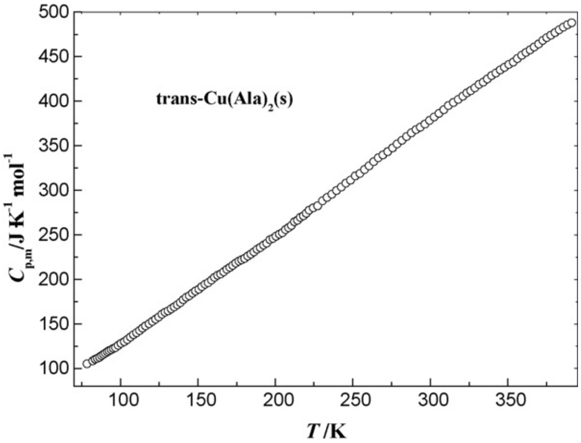

Fig. 1. The curve of the experimental molar heat capacities of the complex *trans*-Cu $(Ala)_2(s)$ vs. the temperature (T).

Table 2
Reaction scheme used to determine the standard molar enthalpy of formation of the complex *trans*-$Cu(Ala)_2(s)$ at 298.15 K

No.	Reactions	Solution	$\Delta_f H^\circ_m$ or $(\Delta_S H^\circ_m \pm \sigma_a)$ (kJ mol^{-1})[b]
1	$\{Cu(Ac)_2{\cdot}H_2O\,(s) + 2Ala\,(s)\} + \text{'s'} =$	A	(6.75 ± 0.04), ΔH_1
2	$2HAc\,(l) + H_2O\,(l) = 2{:}1\ HAc\,(aq)$		-3.58^a, ΔH_2
3	$2{:}1\ HAc\,(aq) + \text{'s'} =$	A_1	$-(1.63 \pm 0.01)$, ΔH_3
4	$Cu(Ala)_2\,(s) + \text{'}A_1\text{'} =$	A'	$-(10.24 \pm 0.08)$, ΔH_4
5	$Cu\,(s) + 4H_2\,(g) + 5/2O_2\,(g) + 4C\,(s) = Cu\,(Ac)_2{\cdot}H_2O\,(s)$		-1193.70 [9], ΔH_5
6	$3C\,(s) + 7/2\,H_2\,(g) + O_2\,(g) + 1/2\,N_2\,(g) = Ala\,(s)$		$-(560.0 \pm 1.7)$ [11], ΔH_6
7	$2H_2\,(g) + O_2\,(g) + 2C\,(s) = HAc\,(l)$		$-(483.52 \pm 0.36)$ [10], ΔH_7
8	$H_2\,(g) + 1/2O_2\,(g) = H_2O\,(l)$		$-(285.83 \pm 0.04)$ [12], ΔH_8
9	$Cu\,(s) + N_2\,(g) + 7H_2\,(g) + 2O_2\,(g) + 6C\,(s) =$ *trans*-$Cu(Ala)_2\,(s)$		$-(1038.6 \pm 3.5)$, ΔH_9

The solvent 's' is 2 mol L^{-1} HCl[a] according to Ref. [8].

[b] $\sigma_a = \sqrt{\sum_{i=1}^{5}(x_i - \bar{x})^2/n(n-1)}$, in which n is the experimental number; x_i , a single value in a set of dissolution measurements; $\bar{x}$, the mean value of a set of measurement results.

3.2. Thermodynamic functions of the complex trans-Cu(Ala)$_2$(s)

The smoothed molar heat capacities and thermodynamic functions of the complex *trans*-$Cu(Ala)_2(s)$ were calculated based on the fitted polynomial of the heat capacities as a function of the reduced temperature (X) according to the following thermodynamic equations:

$$H_{(T)} - H_{(298.15\mathrm{K})} = \int_{298.15\mathrm{K}}^{T} C_{p,m}\,\mathrm{d}T$$

$$S_{(T)} - S_{(298.15\mathrm{K})} = \int_{298.15\mathrm{K}}^{T} C_{p,m}T^{-1}\,\mathrm{d}T$$

The polynomial fitted values of the molar heat capacities and fundamental thermodynamic functions of the sample relative to the standard reference temperature 298.15 K were tabulated in Table 3 (see supplementary data) with the interval of 5 K.

3.3. The determination of enthalpy change for the solid-state coordination reaction of Cu(Ac)$_2$·H$_2$O (s) with alanine

The solid-state coordination reaction of $Cu(Ac)_2{\cdot}H_2O(s)$ with alanine is shown as follows:

$$Cu(Ac)_2{\cdot}H_2O(s) + 2Ala(s) \rightarrow trans\text{-}Cu(Ala)_2(s) + 2HAc(l) + H_2O(l) \quad (1)$$

The enthalpy change of the reaction (1) was determined by measuring enthalpies of dissolution of the $\{Cu(Ac)_2{\cdot}H_2O(s) + 2Ala(s)\}$ and the 2:1 HAc(aq) in 2 mol dm^{-3} hydrochloric acid, and the *trans*-$Cu(Ala)_2{\cdot}H_2O(s)$ in the solvent [2:1 HAc(aq) + 2 mol dm^{-3} HCl] at 298.15 K.

Of about 0.377 g sample of the $\{Cu(Ac)_2{\cdot}H_2O(s)$ and $2Ala(s)\}$ mixture at mole ratio of $n(trans\text{-}Cu(Ac)_2{\cdot}H_2O){:}n(Ala) = 1{:}2$ were dissolved in 100 ml of 2 mol dm^{-3} HCl at 298.15 K.

If ‘s’ = calorimetric solvent, 2 mol dm^{-3} HCl, then,

$$\{Cu(Ac)_2 \cdot H_2O(s) + 2Ala(s)\} + \text{‘s’} = \text{solution} \quad A$$

The dissolution enthalpies of HAc (aq) $[n(HAc)/n(H_2O)] = 2{:}1$ in 100 ml of 2 mol dm^{-3} HCl were measured under the same condition,

$$2 : 1HAc(aq) + \text{‘s’} = \text{solution} \quad A_1.$$

The dissolution enthalpies of *trans*-$Cu(Ala)_2$ (s) in solution A_1 were measured under the same condition as the above,

$$trans\text{-}Cu(Ala)_2(s) + \text{solution} \quad A_1 = \text{solution} \quad A’.$$

The measurement results of dissolution enthalpies for the reactants and products of reaction (1) are listed in Table 1. In addition, the enthalpy change of mixing HAc (l) with H_2O (l) at the mole ratio of $n(HAc)/n(H_2O) = 2{:}1$ can be obtained from the literature [8]: $\Delta_S H_m^\circ = -3.58\ \text{kJ mol}^{-1}$. The enthalpy change of the solid-state coordination reaction (1) can be calculated in accordance with a thermochemical cycle and the experimental results as follows:

$$\begin{aligned}\Delta_r H_m(1) &= \Delta_S H_m^\circ[Cu(Ac)_2 \cdot H_2O(s) + 2Ala(s)] - \Delta_S H_m^\circ[2 : 1\,HAc(aq)]\\ &\quad - \Delta_S H_m^\circ[trans\text{-}Cu(Ala)_2(s)] - \Delta_S H_m^\circ\\ &= (22.20 \pm 0.09)\ \text{kJ mol}^{-1}.\end{aligned}$$

3.4. *The standard molar enthalpy of formation of trans-Cu(Ala)$_2$(s)*

A reaction scheme used to derive the standard molar enthalpy of formation of *trans*-$Cu(Ala)_2(s)$ was given in Table 2. The experimental values of the dissolution enthalpies of the reactants and products in the solid-state coordination reactions (1) were combined with auxiliary thermodynamic data of $\Delta_f H_m^\circ[Cu(E)_2 \cdot H_2O,s] = -1193.70\ \text{kJ mol}^{-1}$ [9], $\Delta_f H_m^\circ(HAc, l) = -(483.52 \pm 0.36)\ \text{kJ mol}^{-1}$ [10], $\Delta_f H_m^\circ(Ala,s) = -(560.0 \pm 1.7)\ \text{kJ mol}^{-1}$ [11], and $\Delta_f H_m^\circ(H_2O, l) = -(285.83 \pm 0.04)\ \text{kJ mol}^{-1}$ [12] to derive the standard molar enthalpy of formation of *trans*-$Cu(Ala)_2$ (s),

$\Delta_f H_m^\circ[trans\text{-}Cu(Ala)_2, s] = \Delta_r H_m(1) + \Delta_f H_m^\circ[Cu(Ac)_2 \cdot H_2O, s] + 2\Delta_f H_m^\circ(Ala, s) - 2\Delta_f H_m^\circ(HAc, l) - \Delta_f H_m^\circ(H_2O, l) = \Delta H_9 = \Delta H_1 - \Delta H_2 - \Delta H_3 - \Delta H_4 + \Delta H_5 + 2\Delta H_6 - 2\Delta H_7 - \Delta H_8 = -(1038.6 \pm 3.5)\text{kJ mol}^{-1}$, in which $\Delta H_1 \sim \Delta H_9$ are the enthalpy changes of the reactions corresponding to No. of the reaction in Table 1. The uncertainty $\pm 3.5\ \text{kJ mol}^{-1}$ was calculated according to the error transfer formula, such as for the equations, $y = ax + bz$, or $y = ax - bz$, total error (R) of y equals the algebraic sum of errors of x and z, that is, $R = \sqrt{a^2 r_x^2 + b^2 r_y^2}$, in which a and b are the constants, r_x and r_y are the errors of the variables x and y.

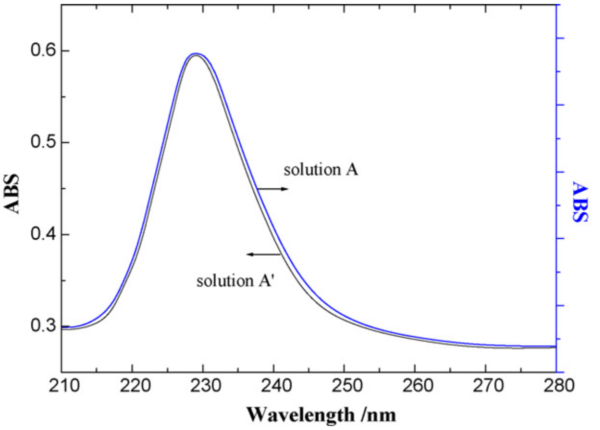

Fig. 2. UV–vis spectra of solution A and solution A’ obtained from the dissolution of the $\{Cu(Ac)_2 \cdot H_2O(s) + 2Ala(s)\}$ mixture in 100 cm^3 of 2 mol dm^{-3} HCl and the $\{trans\text{-}Cu(Ala)_2(s)\}$ in the solution A_1 (diluted into 1:20).

The results of UV–vis spectrum and refrangibility (refractive index) were two important information used to detect whether the structure and composition of a kind of solution was the same or not as another. In this paper, all of the reactants and products of the reaction (1) can be easily dissolved in the selected solvent. The measured values of the refractive indexes of solution A and solution A’ were (1.2786 ± 0.0011) and (1.2782 ± 0.0008), respectively. The results of UV–vis spectroscopy were shown in Fig. 2. UV–vis spectrum and the data of the refractive indexes of solution A obtained agreed with those of solution A’, no difference in the structure and chemical composition existed between the two solutions. These results have demonstrated that the physicochemical properties of the solutions A were the same as those of the solution A’.

4. Conclusions

(1) The paper reports low-temperature heat capacities measured by adiabatic calorimetry and the dissolution enthalpies of the reactants and the products of the solid-state coordination reaction of copper(II) acetate with L-α-alanine by isoperibol solution calorimetry. Additionally, the thermodynamic functions and standard molar enthalpy of formation of the product [*trans*-$Cu(Ala)_2(s)$] were derived from these experimental results.

(2) The reliability of the designed thermochemical cycle has been verified by UV spectroscopy and the data of the refractive indexes. It is shown that the cycle is reasonable and can be used to determine standard molar enthalpy of formation of the product [*trans*-$Cu(Ala)_2(s)$]. The uncertainty of the standard molar enthalpy of formation obtained by isoperibol solution calorimetry was estimated to be between 0.3% and 0.5%, chiefly considering the measurements of voltage changes E_s and E_e, duration time of electric calibration t, final data processing and so on.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tca.2008.02.024.

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