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Standard molar enthalpies of formation of $Ni(CH_3COO)_2$, $Ni(CH_3COO)_2 \cdot 4.00H_2O$, $Cd(CH_3COO)_2$, and $Cd(CH_3COO)_2 \cdot 2.00H_2O$ in the crystalline state

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The standard ($p^{\circ} = 0.1$ MPa) molar enthalpies of formation at the temperature T = 298.15 K of crystalline nickel(II) acetate, Ni(CH₃COO)₂, nickel(II) acetate tetrahydrate Ni(CH₃COO)₂ · 4.00H₂O, cadmium(II) acetate Cd(CH₃COO)₂, and cadmium(II) acetate dihydrate Cd(CH₃COO)₂ · 2.00H₂O were determined by solution-reaction calorimetry. The enthalpies of dehydration were derived for both complexes.

	$\Delta_{\rm f} H_{\rm m}^{\rm o}({\rm cr})/({\rm kJ}\cdot{\rm mol}^{-1})$
$\frac{\text{Ni}(\text{CH}_3\text{COO})_2(\text{cr})}{\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4.00\text{H}_2\text{O}(\text{cr})}$ $\frac{\text{Cd}(\text{CH}_3\text{COO})_2(\text{cr})}{\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2.00\text{H}_2\text{O}(\text{cr})}$	$\begin{array}{r} -961.5 \pm 1.7 \\ -2168.2 \pm 1.0 \\ -1026.3 \pm 1.4 \\ -1622.2 \pm 1.2 \end{array}$

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KEYWORDS: enthalpies of formation; nickel acetate; cadmium acetate; differential scanning calorimetry; solution-reaction calorimetry; enthalpies of dehydration

1. Introduction

The anhydrous and hydrated acetates of several metals are widely used as reagents for the preparation of metal complexes, not only because they have good solubility in a wide range of organic and mixed solvents, but mainly because of the buffer capability of the metallic acetates which facilitates the synthesis of the respective metal complexes. Therefore, these metal acetates are obviously convenient reagents to be used in the thermochemical reactions which are the bases of the determination of the standard molar enthalpies of

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	$10^2 \cdot w(C)$	Calculated $10^2 \cdot w(H)$	$10^2 \cdot w(M)$	$10^2 \cdot w(C)$	Found $10^2 \cdot w(H)$	$10^2 \cdot w(M)$
Ni(CH ₃ COO) ₂	27.18	3.42	33.20	27.40	3.28	
$Ni(CH_3COO)_2 \cdot 4.00H_2O$	19.31	5.67	23.59	19.00	5.64	23.66
$Cd(CH_3COO)_2$	20.84	2.62	48.77	20.58	2.60	
$Cd(CH_3COO)_2 \cdot 2.00H_2O$	18.03	3.78	42.18	17.55	3.68	42.09

TABLE 1. Mass fraction w of elements in the complexes

formation $\Delta_f H_m^o$ of metal complexes by reaction and solution calorimetry. This implies a knowledge of precise and accurate values for the standard molar enthalpies of formation of the respective metal acetates.

In the literature⁽¹⁾ there are reported values for the standard molar enthalpies of formation, at T = 298.15 K in the crystalline state, only for the anhydrous nickel(II) and cadmium(II) acetates, $-(966 \pm 17)$ kJ \cdot mol⁻¹ and -966 kJ \cdot mol⁻¹, respectively. For the cadmium acetate there is no reported associated uncertainty and for the nickel acetate the associated uncertainty is too large, which limits the usefulness of these compounds in appropriate reactions for the determination of the standard molar enthalpies of formation of other chemical species. In order to overcome this difficulty, we determined by high precision solution and reaction calorimetry, and report in this paper, experimental values for the standard molar enthalpies of formation, at T = 298.15 K, of crystalline Ni(CH₃COO)₂, Ni(CH₃COO)₂ \cdot 4.00H₂O, Cd(CH₃COO)₂, and Cd(CH₃COO)₂ \cdot 2.00H₂O.

2. Experimental

The nickel(II) and cadmium(II) salts, Ni(CH₃COO)₂ · 4H₂O, Cd(CH₃COO)₂ · 2H₂O, NiCl₂ · 6H₂O and CdSO₄ · 8/3H₂O, all Merck *p.a.*, were maintained in desiccators over silica gel until the exact proportion of water of hydration was achieved, as checked by EDTA volumetric analyses of the metal cation. The purity of all the compounds was checked by elemental carbon and hydrogen microanalyses.

Anhydrous Ni(CH₃COO)₂ and Cd(CH₃COO)₂ were obtained from the hydrated analogues by heating under reduced pressure (p < 10 Pa), at the temperature 390 K for 2 d. The purities of the anhydrous nickel and cadmium acetates were checked by elemental analyses and F.t.i.r., where no O–H bonds were detected; the mass fractions w of C, H, and metal were as shown in table 1. All the manipulations of the metal acetates were done in a glove box under a nitrogen atmosphere.

The calorimetric solvents, HCl (aq, $c = 1.000 \text{ mol} \cdot \text{dm}^{-3}$), H₂SO₄ (aq, $c = 0.500 \text{ mol} \cdot \text{dm}^{-3}$) and CH₃COOH (aq, $c = 2.000 \text{ mol} \cdot \text{dm}^{-3}$) were obtained by dilution with deionized water of Merck Titrisol ampoules of the acids: the compositions were,⁽²⁾ respectively, HCl \cdot 54.36H₂O, H₂SO₄ \cdot 108.92H₂O, and CH₃COOH \cdot 54.36H₂O.

All the compounds were studied in a Dewar isoperibol solution-reaction calorimeter^(3,4) with a newly designed all-glass reaction vessel⁽⁵⁾ (containing 120.0 cm³ of solvent),

equipped with a twin-bladed stirrer and with an ampoule holder that allows the introduction of new ampoules into the reaction vessel without having to take the reaction vessel out of the thermostatic bath. The calorimeter temperatures were measured using a quartz crystal thermometer (Hewlett Packard HP 2804-A) to 10^{-4} K, every 10 s. The calorimetric system was interfaced to a PC using the program LABTERMO⁽⁵⁾ to control the system. The vessel was submerged in a water thermostatic bath maintained at $T = (298.150 \pm 0.001)$ K by a TRONAC PTC-41 controller. The accuracy and performance of the calorimeter were tested by measuring the molar enthalpy of solution of THAM tris(hydroxymethyl)aminomethane (BDH, Thermochemical Standard) in 0.100 mol \cdot dm⁻³ HCl(aq) at T = 298.15 K. The value obtained: $\Delta_r H_m^o = -(29.742 \pm 0.029)$ kJ \cdot mol⁻¹ (mean of ten experiments) is in agreement with the literature value of Kilday and Prosen⁽⁶⁾ $-(29.770 \pm 0.032)$ kJ \cdot mol⁻¹.

The differential scanning calorimetric measurements were carried out in a heat power compensation d.s.c. type, model DSC 141 from Setaram. The crucibles containing the sample and the empty crucibles used as the reference were made of aluminium and had a volume of 30 mm³. The calibrations of the temperature and heat flux scales of the calorimeter were performed by Setaram; the temperature scale was calibrated by measuring the melting temperatures of Hg, In, Sn, Pb, and Zn, at different heating rates, and the heat flux scale was calibrated by the Joule effect using the same standards. Measurements of the temperature of fusion and of the enthalpy of fusion of benzoic acid (NIST SRC 39i) were made in our laboratory to check the accuracy of the instrument.

The relative atomic masses were those recommended by the IUPAC Commission in 1995.⁽⁷⁾ All uncertainty intervals given are twice the standard deviation of the mean. Carbon and hydrogen analyses were carried out in the Microanalytical Unit of the Chemistry Department in Porto.

3. Results

The thermochemical reaction for determining the standard molar enthalpy of formation of $Ni(CH_3COO)_2 \cdot 4.00H_2O(cr)$ was:

$$57.08H_2O(l) + 2CH_3COOH \cdot 24.82H_2O(aq) + NiCl_2 \cdot 6H_2O(cr)$$

= 2HCl \cdot 54.36H_2O(aq) + Ni(CH_3COO)_2 \cdot 4.00H_2O(cr). (1)

The calorimetric solvent was HCl \cdot 54.36H₂O(aq). The standard enthalpy $\Delta_r H_m^o$ of thermochemical reaction (1) was determined by successively measuring the enthalpies of solution and reaction $\Delta_i H_m$ of stoichiometric amounts of each reactant and product in the calorimetric solvent ($v = 120.0 \text{ cm}^3$), so that the final solution resulting from the dissolution of all the reactants was of the same composition as that from the dissolution of all the products. As a check of the validity of this procedure, ampoules of the final solution of the dissolution of all products in the calorimeter and no enthalpy change was detected. Table 2 lists the mean values of the molar enthalpies of solution and reaction required for the determination of the enthalpy of the thermochemical reaction (1), according to:

$$\Delta_{\rm r} H_{\rm m}({\rm l}) = 57.08 \cdot \Delta_1 H_{\rm m} + 2 \cdot \Delta_2 H_{\rm m} + \Delta_3 H_{\rm m} - 2 \cdot \Delta_4 H_{\rm m} - \Delta_5 H_{\rm m} = (2.79 \pm 0.91) \,\rm kJ \cdot mol^{-1}$$
(2)

 $\Delta_{i}H_{m}/(kJ \cdot mol^{-1})$ Reactant Solvent Solution No. of i formed expts $HCl \cdot 54.36H_2O(aq)$ -0.005 ± 0.002^{a} 1 $H_2O(l)$ A_1 2 $CH_{3}COOH \cdot 24.82H_{2}O(aq)$ -0.0188 ± 0.0010 A_1 A_2 5 A3 3 $NiCl_2 \cdot 6.00H_2O(cr)$ A_2 5 10.33 ± 0.86 4 $HCl \cdot 54.36H_2O(aq)$ $HCl \cdot 54.36H_2O(aq)$ 0.00 B_1 5 Ni(CH₃COO)₂ · 4.00H₂O(cr) 6 4.53 ± 0.36 B_1 A₃

TABLE 2. Molar enthalpies of solution and reaction (T = 298.15 K) $\Delta_i H_m$ for Ni(CH₃COO)₂ · 4.00H₂O(cr)

^a Interpolated from literature data.⁽⁹⁾

TABLE 3. Molar enthalpies of solution and reaction (T = 298.15 K) $\Delta_i H_m$ for Ni(CH₃COO)₂(cr)

i	Reactant	Solvent	Solution	No. of expts	$\Delta_{\rm i} H_{\rm m}/({\rm kJ}\cdot{\rm mol}^{-1})$
1	H ₂ O(l)	$H_2O(l)$	A ₁		0.00
2	Ni(CH ₃ COO) ₂ (cr)	A_1	A ₂	5	-60.42 ± 0.32
3	$\rm Ni(CH_3COO)_2 \cdot 4.00H_2O(cr)$	$\mathrm{H}_{2}\mathrm{O}(\mathrm{l})$	A ₂	5	2.96 ± 0.76

The Ni(CH₃COO)₂(cr) was studied using thermochemical reaction (3) for which the calorimetric solvent was $H_2O(l)$:

$$4H_2O(1) + Ni(CH_3COO)_2(cr) = Ni(CH_3COO)_2 \cdot 4.00H_2O(cr).$$
(3)

Table 3 lists the mean values of the molar enthalpies of solution and reaction required for the determination of the enthalpy of thermochemical reaction (3), according to:

$$\Delta_{\rm r} H_{\rm m}(3) = 4 \cdot \Delta_1 H_{\rm m} + \Delta_2 H_{\rm m} - \Delta_3 H_{\rm m} = -(63.38 \pm 0.82) \,\,\rm kJ \cdot mol^{-1}. \tag{4}$$

The $Cd(CH_3COO)_2 \cdot 2.00H_2O(cr)$ was studied using thermochemical reaction (5) with the calorimetric solvent being $H_2SO_4 \cdot 108.92H_2O(aq)$:

$$58.61H_2O(1) + 2CH_3COOH \cdot 24.82H_2O(aq) + CdSO_4 \cdot (8/3)H_2O(cr)$$

= H_2SO_4 \cdot 108.92H_2O(aq) + Cd(CH_3COO)_2 \cdot 2.00H_2O(cr). (5)

Table 4 lists the mean values of the molar enthalpies of solution and reaction required for the determination of the enthalpy of thermochemical reaction (5), according to:

$$\Delta_{\rm r} H_{\rm m}(5) = 58.61 \cdot \Delta_1 H_{\rm m} + 2 \cdot \Delta_2 H_{\rm m} + \Delta_3 H_{\rm m} - \Delta_4 H_{\rm m} - \Delta_5 H_{\rm m} = (15.89 \pm 0.57) \,\text{kJ} \cdot \text{mol}^{-1}.$$
(6)

$\Delta_{\rm f} H_{\rm m}^{\rm o}({\rm cr})$ of nickel and cadmium acetates

TABLE 4. Molar enthalpies of solution and reaction (T = 298.15 K) $\Delta_i H_m$ for $Cd(CH_3COO)_2 \cdot 2.00H_2O(cr)$

i	Reactant	Solvent	Solution formed	No. of expts	$\Delta_i H_m/(kJ \cdot mol^{-1})$
1	H ₂ O(l)	$H_2SO_4\cdot 108.92H_2O(aq)$	A ₁		-0.0117 ± 0.0010^{a}
2	$CH_{3}COOH \cdot 24.82H_{2}O(aq)$	A ₁	A ₂	5	0.00 ± 0.30
3	$CdSO_4\cdot 8/3H_2O(cr)$	A ₂	A ₃	5	-1.51 ± 0.15
4	$H_2SO_4\cdot 108.92H_2O(aq)$	$H_2SO_4\cdot 108.92H_2O(aq)$	B_1		0.00
5	$Cd(CH_3COO)_2 \cdot 2.00H_2O(cr)$	B ₁	A ₃	5	-18.09 ± 0.39

^a Interpolated from literature data.⁽⁹⁾

TABLE 5. Molar enthalpies of solution and reaction (T = 298.15 K) $\Delta_i H_m$ for $Cd(CH_3COO)_2(cr)$

i	Reactant	Solvent	Solution	No. of expts	$\Delta_{\rm i} H_{\rm m}/({\rm kJ}\cdot{\rm mol}^{-1})$
1	H ₂ O(l)	$H_2O(l)$	A ₁		0.00
2	$Cd(CH_3COO)_2(cr)$	A_1	A ₂	5	-33.17 ± 0.57
3	$Cd(CH_3COO)_2 \cdot 2.00H_2O(cr)$	$\mathrm{H}_{2}\mathrm{O}(l)$	A ₂	5	-9.06 ± 0.42

The $Cd(CH_3COO)_2(cr)$ was studied using thermochemical reaction (7) with H_2O as the calorimetric solvent:

$$2H_2O(l) + Cd(CH_3COO)_2(cr) = Cd(CH_3COO)_2 \cdot 2.00H_2O(cr).$$
(7)

Table 5 lists the mean values of the molar enthalpies of solution and reaction required for the determination of the enthalpy of thermochemical reaction (7), according to:

$$\Delta_{\rm r} H_{\rm m}(7) = 2 \cdot \Delta_1 H_{\rm m} + \Delta_2 H_{\rm m} - \Delta_3 H_{\rm m} = -(24.11 \pm 0.73) \,\rm kJ \cdot mol^{-1}.$$
(8)

To derive the standard molar enthalpies of formation of the crystalline metal acetates, the following auxiliary quantities, at T = 298.15 K, were used: $\Delta_f H_m^0(H_2O, I) = -(285.830 \pm 0.042)$ kJ·mol⁻¹;⁽⁸⁾ $\Delta_f H_m^0(NiCl_2 \cdot 6.00H_2O, cr) = -(2103.17 \pm 0.21)$ kJ·mol⁻¹;⁽⁹⁾ $\Delta_f H_m^0(HCl in 54.36H_2O, aq) = -(165.42 \pm 0.01)$ kJ·mol⁻¹;⁽⁹⁾ $\Delta_f H_m^0(CdSO_4 \cdot 8/3H_2O, cr) = -(1729.4 \pm 1.0)$ kJ·mol⁻¹;⁽¹⁰⁾ $\Delta_f H_m^0(H_2SO_4 in 108.92 H_2O, aq) = -(887.74 \pm 0.01)$ kJ·mol⁻¹;⁽⁹⁾ $\Delta_f H_m^0(CH_3COOH in 24.82H_2O, aq) = -(485.16 \pm 0.01)$ kJ·mol⁻¹;⁽⁹⁾ the values of the standard molar enthalpies of formation of Ni(CH_3COO)_2 \cdot 4.00H_2O(cr) and Cd(CH_3COO)_2 \cdot 2.00H_2O(cr) derived in the present work have been used in the calculation for the anhydrous acetates. Table 6 lists the derived standard molar enthalpies of formation of the crystalline hydrated and anhydrous Ni(II) and Cd(II) acetates.

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TABLE 6. Standard molar enthalpies of reaction $\Delta_r H_m^o$ and formation of $\Delta_f H_m^o$ of the crystalline metal acetates

 $\Delta_{\rm r} H_{\rm m}^{\rm o}/({\rm kJ}\cdot{\rm mol}^{-1})$ $\Delta_{\rm f} H_{\rm m}^{\rm o}/({\rm kJ}\cdot{\rm mol}^{-1})$ Complex Literature⁽¹⁾ This work $Ni(CH_3COO)_2 \cdot 4.00H_2O(cr)$ 2.79 ± 0.91 -2168.2 ± 1.0 Ni(CH₃COO)₂(cr) -63.38 ± 0.82 -961.5 ± 1.7 -966 ± 17 Cd(CH₃COO)₂ · 2.00H₂O(cr) 15.89 ± 0.59 -1622.0 ± 1.2 Cd(CH₃COO)₂(cr) -24.11 ± 0.73 -1026.3 ± 1.4 -966

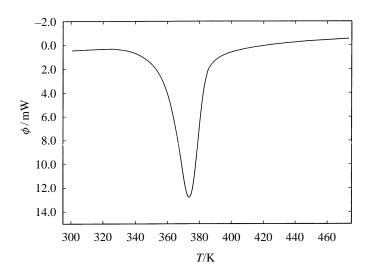


FIGURE 1. Heat flow rate ϕ against temperature T for Ni(CH₃COO)₂ · 4.00H₂O. The peak indicates an endothermic reaction.

Thermograms of Ni(CH₃COO)₂ · 4.00H₂O(cr) and Cd(CH₃COO)₂ · 2.00H₂O(cr) were obtained on heating the compounds at a rate of 0.01667 K · s⁻¹ from room temperature to 470 K. Figure 1 shows the result obtained for Ni(CH₃COO)₂ · 4.00H₂O(cr) which exhibits an endothermic peak in the temperature range 350 K–385 K, corresponding to the loss of four molecules of water for each molecule of nickel(II) acetate; the loss of water was confirmed by the loss of mass of the materials in the crucibles after each run. This behaviour is in agreement with the crystal structure of Ni(CH₃COO)₂ · 4.00H₂O⁽¹⁰⁾ in which four water molecules are coordinated to the metal in equivalent positions.

Figure 2 shows the results obtained for the $Cd(CH_3COO)_2 \cdot 2.00H_2O(cr)$, which exhibits two endothermic peaks, the first one for the temperature range 345 K–365 K and the second one for the 375 K–420 K temperature interval corresponding to the dehydration of the cadmium acetate. The losses of mass of the crucibles and contents

 $\Delta_{\rm f} H_{\rm m}^{\rm o}({\rm cr})$ of nickel and cadmium acetates

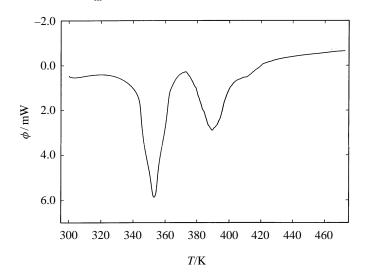


FIGURE 2. Heat flow rate ϕ against temperature T for Cd(CH₃COO)₂ · 2.00H₂O. The peaks indicate endothermic reactions.

after each run are in agreement with the loss of two molecules of water for each molecule of cadmium(II) acetate. Since the enthalpy changes calculated from each peak are equal within the experimental uncertainty, the presence of these two peaks shows that the loss of the two water molecules occurs stepwise. This is accordance with the crystal structure of $Cd(CH_3COO)_2 \cdot 2.00H_2O$,⁽¹¹⁾ where the two water molecules are bound in non-equivalent positions around the metal, the two cadmium–oxygen bond distances being significantly different: 0.2299 nm and 0.2325 nm.⁽¹¹⁾

In the case of Ni(CH₃COO)₂ · 4.00H₂O(cr), five experiments gave the average value of $(229.4 \pm 6.1) \text{ kJ} \cdot \text{mol}^{-1}$ for the enthalpy of dehydration at the temperature of the experiments, whereas in the case of Cd(CH₃COO)₂ · 2.00H₂O(cr) the average of five experiments yielded the value of $(105.4 \pm 4.1) \text{ kJ} \cdot \text{mol}^{-1}$ for the dehydration of this compound, also at the temperature of the experiments. The lack of heat capacity values for the species involved in these dehydration processes does not allow the calculation of the dehydration enthalpies at T = 298.15 K.

4. Discussion

From the standard molar enthalpies of formation of the anhydrous and hydrated Ni(II) and Cd(II) acetates at T = 298.15 K determined in this work, the values of standard molar enthalpies of dehydration at T = 298.15 K, $\Delta_r H_m^o$ of the hydrated complexes can be calculated from reactions (9) and (10), with $\Delta_f H_m^o$ (H₂O, g) = $-(241.826 \pm 0.040)$ kJ · mol⁻¹:⁽⁸⁾

$$Ni(CH_3COO)_2 \cdot 4.00H_2O(cr) = Ni(CH_3COO)_2(cr) + 4H_2O(g),$$
 (9)

 $\Delta_{\rm r} H_{\rm m}^{\rm o} = (239.4 \pm 2.0) \, \rm kJ \cdot mol^{-1};$

$$Cd(CH_3COO)_2 \cdot 2.00H_2O(cr) = Cd(CH_3COO)_2(cr) + 2H_2O(g),$$
 (10)

 $\Delta_{\rm r} H_{\rm m}^{\rm o} = (112.2 \pm 1.8) \, \rm kJ \cdot mol^{-1}.$

These calculated values of the dehydration enthalpies of the metal acetates at T = 298.15 K are in reasonable agreement with those measured by d.s.c. at higher temperatures.

The only literature⁽¹⁾ value for $\Delta_f H_m^o \{ Ni(CH_3COO)_2, cr \} = -(966 \pm 17) \text{ kJ} \cdot \text{mol}^{-1} \text{ was obtained by Van My and Perinet⁽¹²⁾ in the late 1960s, by Tian–Calvet microcalorimetry. It is in reasonable agreement with our solution calorimetry value, but has a large uncertainty associated with it, which would induce other larger uncertainties when measuring other thermochemical properties using a reaction in which this compound takes part.$

For the Cd(CH₃COO)₂ · 2.00H₂O(cr), the literature^(1,13) value of $\Delta_f H_m^o(cr) = -966 \text{ kJ} \cdot \text{mol}^{-1}$ was first reported in 1959 by Morris from calculations based on lyotropic numbers and enthalpies of hydration of metal ions together with the enthalpy of formation of the acetate radical, and has no uncertainty associated with it. This value is considerably different from the experimental one we obtained by solution calorimetry, which seems to be a more reliable method.

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