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Phase Equilibrium System of $LuCl_3-CdCl_2-HCl(7.47\ \%)-H_2O$ at 298.15 K and Standard Molar Enthalpies of Formation of New Solid-Phase Compounds

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The equilibrium solubility of the quaternary system $LuCl_3-CdCl_2-HCl-H_2O$ was determined at 298.15 K, and the corresponding equilibrium diagram was constructed in this paper. The quaternary system is complicated with four equilibrium solid phases, $CdCl_2\cdot H_2O$, $Cd_3LuCl_9\cdot 14H_2O$ (3:1 type), $CdLuCl_5\cdot 9H_2O$ (1:1 type), and $LuCl_3\cdot 6H_2O$, of which the new compounds $Cd_3LuCl_9\cdot 14H_2O$ and $CdLuCl_5\cdot 9H_2O$ were found to be congruently soluble in the system. The two new compounds obtained were identified and characterized by the methods of X-ray diffraction, thermogravimetry, and differential thermogravimetry. The standard molar enthalpies of solution of $Cd_3LuCl_9\cdot 14H_2O$ and $CdLuCl_5\cdot 9H_2O$ in deionized water were measured to be $28.79 \pm 0.22\ kJ\cdot mol^{-1}$ and $-15.48 \pm 0.30\ kJ\cdot mol^{-1}$, respectively, by heat conduction microcalorimetry. Their standard molar enthalpies of formation were calculated to be $-6427.5 \pm 1.3\ kJ\cdot mol^{-1}$ and $-4133.7 \pm 0.7\ kJ\cdot mol^{-1}$, respectively.

Introduction

Phase equilibria of quaternary systems of $RECl_3-CdCl_2-HCl(8\ \%)-H_2O$ (RE = La, Ce, Pr, Nd, Eu, Dy, Y, Er) at 298.15 K have been previously reported.[1–8] Sixteen new compounds were obtained from the systems. They are $Cd_9RECl_{21}\cdot nH_2O$ (RE = Ce, Nd, Eu), $Cd_9RE_2Cl_{24}\cdot nH_2O$ (RE = Dy, Er), $Cd_8RECl_{19}\cdot nH_2O$ (RE = La, Pr), $Cd_6CeCl_{15}\cdot 14H_2O$, $Cd_5NdCl_{13}\cdot 14H_2O$, $Cd_5Y_2Cl_{16}\cdot 26H_2O$, $Cd_4RECl_{11}\cdot nH_2O$ (RE =La, Ce, Pr, Eu, Y), and $CdEr_7Cl_{23}\cdot 42H_2O$. Comparison of the quaternary systems finds that phase chemical reactions of the two quaternary systems $RECl_3-CdCl_2-HCl-H_2O$ (RE = La, Pr) are very similar because they all have the 8:1 type and 4:1 type compounds, although for the three quaternary systems $RECl_3-CdCl_2-HCl-H_2O$ (RE = Ce, Eu, Nd), their phase chemical reactions have both similarities (all having a 9:1 type compound) and dissimilarities (all having various types of compounds). The quaternary system $YCl_3-CdCl_2-HCl-H_2O$ and the four quaternary systems $RECl_3-CdCl_2-HCl-H_2O$ (RE = La, Ce, Pr, Eu) were compared further, where it was found that their phase chemical reactions also have both similarities (all having a 4:1 type compound) and dissimilarities. Similarly, the quaternary systems $RECl_3-CdCl_2-HCl-H_2O$ (Dy, Er) have both similarities and dissimilarities.

To contrast the difference of the phase chemical relationship in $LuCl_3-CdCl_2-HCl-H_2O$ with the $RECl_3-CdCl_2-HCl-H_2O$ (RE = La, Ce, Pr, Nd, Eu, Dy, Y, Er) systems and find more phase equilibrium information on the $RECl_3-CdCl_2-HCl-H_2O$ quaternary systems, it is necessary for us to further study the phase equilibria systematically. The present paper is concerned with the solubility and phase equilibrium relation of the $LuCl_3-CdCl_2-HCl(7.47\ \%)-H_2O$ system at 298.15 K. In this system, two new solid-phase compounds were found, and related measurement of their properties was also done.

Experimental Section

Preparing Samples. All chemicals ($CdCl_2$) and solvents [H_2O and HCl (37 mass %)] were analytically pure and purchased from the market. $LuCl_3\cdot 6H_2O$ was prepared by the reaction of Lu_2O_3 (99.99 mass %) with hydrochloric acid (37 mass % HCl). The composition of $LuCl_3\cdot 6H_2O$ was confirmed by analyzing the Cl^- content by titration with a normal solution of silver nitrate and the Lu^{3+} content by titration with EDTA. The purity reached in this way was found to be 99.9 mass %. The analysis errors for those ions were relative ones and better than ± 0.21 %.

Investigations on the System at 298.15 K and Analysis Methods. The method of the investigation of the solubility of the $LuCl_3-CdCl_2-HCl(7.47\ \%)-H_2O$ quaternary system has been previously reported.[3] All sealed samples were put in a big water tank with a thermostat fixed at 298.15 K and an electrical stirrer. The precision of the temperature was 0.1 K. The solid–liquid-phase equilibrium was established for these samples after 10 to 12 days.

The saturated solutions and the corresponding wet solid phases (wet residues) of the samples were separated, taken out, and analyzed. The analysis methods were as follows: (1) the concentration of protons was analyzed by titration with a solution of sodium hydroxide; (2) Lu^{3+} by titration with a normal solution of EDTA after Cd^{2+} was blanketed with a screening agent of 1,10-phenanthroline; (3) the total amount of Cl^- by titration with a normal solution of silver nitrate; (4) the total amount of Cl^- minus the amount of Cl^- combined with Lu^{3+} and H^+ is that of Cl^- combined with Cd^{2+}. The amount of Cd^{2+} is equal to the half-amount of Cl^- combined with Cd^{2+}. The composition of the saturated solution and the corresponding wet solid-phase points were determined by calculating the individual contents of HCl, $LuCl_3$, and $CdCl_2$ according to the analysis results of the H^+, Lu^{3+}, and Cl^- ions. The solid-phase compositions in the system were determined graphically by the well-known wet residue method of Schreinemakers[9] and checked by chemical analysis. The results of analyses of each sample for the quaternary system are shown

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Table 1. Solubility Data of the Saturated Solution of the Quaternary System $CdCl_2-LuCl_3-HCl$(7.47 mass %)$-H_2O$ at (298 ± 0.1) K and Central Projection Data on the Trigonal Basal Face $CdCl_2-LuCl_3-H_2O$

	composition of solution (% mass weight)					composition of residue (% mass weight)					
	composition in the tetrahedral			composition in the trigonal basal face[a]		composition in the tetrahedral			composition in the trigonal basal face		
no.	HCl	$CdCl_2$	$LuCl_3$	$CdCl_2$	$LuCl_3$	HCl	$CdCl_2$	$LuCl_3$	$CdCl_2$	$LuCl_3$	solid phase[b]
1	8.71	47.62	0	52.16	0	---	---	---	---	---	A
2	7.94	46.21	2.65	50.19	2.88	4.15	67.53	1.61	70.45	1.68	A
3	7.75	46.77	4.31	50.70	4.67	4.38	66.73	2.27	69.79	2.37	A
4	7.19	46.44	6.46	50.04	6.96	3.75	63.20	9.05	65.66	9.40	A+B
5	7.27	46.46	6.59	50.10	7.11	2.11	59.28	13.44	60.56	13.73	A+B
6	7.14	45.99	7.01	49.53	7.55	2.06	49.04	19.94	50.07	20.36	B
7	6.40	42.55	9.90	45.46	10.58	1.78	48.12	21.60	48.99	21.99	B
8	7.76	39.30	10.20	42.61	11.06	1.71	47.65	22.26	48.48	22.65	B
9	7.03	37.62	12.39	40.46	13.33	1.57	47.80	22.82	48.56	23.18	B
10	7.31	34.56	14.56	37.28	15.71	1.64	46.72	23.15	47.50	23.53	B
11	8.45	32.15	16.06	35.12	17.54	1.44	46.92	24.03	47.60	24.38	B
12	8.15	30.94	18.75	33.68	20.41	1.59	46.47	24.13	47.22	24.52	B
13	7.92	30.07	19.96	32.66	21.68	1.33	46.68	24.72	47.31	25.05	B
14	7.60	29.74	22.09	32.19	23.91	2.62	43.10	24.24	44.26	24.89	B
15	7.42	28.94	23.17	31.26	25.03	1.60	45.23	25.06	45.96	25.47	B
16	6.95	29.33	24.47	31.52	26.30	1.88	36.27	32.62	36.96	33.24	B+C
17	6.18	27.80	26.43	29.63	28.17	2.16	29.03	38.40	29.67	39.25	C
18	7.13	24.67	26.89	26.56	28.95	2.50	27.68	38.41	28.39	39.39	C
19	6.60	20.10	30.45	21.52	32.60	2.74	25.11	39.18	25.82	40.28	C
20	8.31	15.96	30.90	17.41	33.70	1.92	25.24	40.73	25.73	41.53	C
21	7.36	13.11	34.65	14.15	37.40	2.33	23.94	41.47	24.51	42.46	C
22	7.72	13.05	34.06	14.14	36.91	1.55	6.60	60.97	6.70	61.93	C+D
23	7.42	10.77	35.49	11.63	38.33	1.61	2.22	64.22	2.26	65.27	D
24	7.44	6.49	37.74	7.01	40.77	1.74	1.65	64.07	1.68	65.20	D
25	7.70	0	40.58	0	43.96	---	---	---	---	---	D

[a] Double saturation point (average): E_1, $CdCl_2$ 50.07 %, $LuCl_3$ 7.03 %; E_2, $CdCl_2$ 31.52 %, $LuCl_3$ 26.30 %; E_3, $CdCl_2$ 14.14 %, $LuCl_3$ 36.91 %. [b] Compounds: A, $CdCl_2 \cdot H_2O$; B, $Cd_3LuCl_9 \cdot 14H_2O$; C, $CdLuCl_5 \cdot 9H_2O$; D, $LuCl \cdot 6H_2O$.

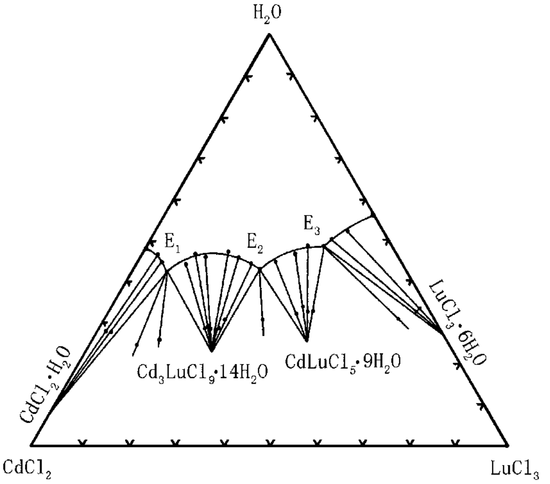

Figure 1. Solubility diagram of the quaternary system $LuCl_3-CdCl_2-HCl$(7.47 mass %)$-H_2O$ on the $LuCl_3-CdCl_2-H_2O$ trigonal basal face at 298.15 K.

in Table 1. As shown in Table 1, the HCl mass percentage in the liquid phase of the quaternary system changes between (6.18 and 8.71) mass %.

Equipment and Conditions. Thermal characterization of the new compounds was undertaken with an SDT Q600 V8.0 thermal analysis apparatus (TG-DTG) that worked with a heating rate of 10 $K \cdot min^{-1}$ under an N_2 atmosphere with a flow rate of 100 $cm^3 \cdot min^{-1}$; X-ray diffraction (XRD) measurements were performed by a D/Max-3C diffractometer using Cu Kα radiation, 50 kV and 80 mA, at room temperature, in air.

All the enthalpies of solution were measured using an RD496-III-2000 heat conduction microcalorimeter (Southwest Institute of Electron Engineering, China), which is a totally automatic instrument utilizing computer control. The microcalorimeter is described in the literature.[10–12]

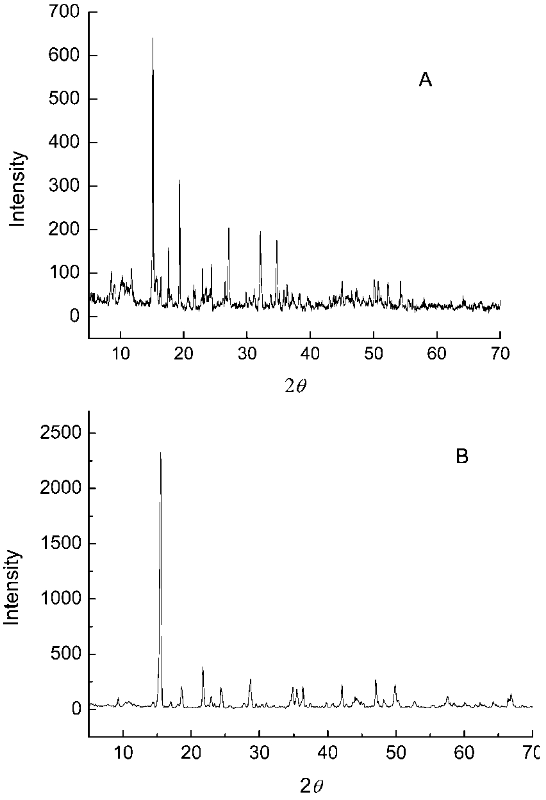

Figure 2. X-ray powder diffraction spectra of $Cd_3LuCl_9 \cdot 14H_2O$ (A) and $CdLuCl_5 \cdot 9H_2O$ (B).

To check the performance of the RD496-III-2000 heat conduction microcalorimeter, the calorimetric constant was determined at 298.15 K by the Joule effect before the experiments. The Joule cell was supplied with 100 mW electric power for 100 s. The calorimetric constant obtained in this way was (59.31 ± 0.04)

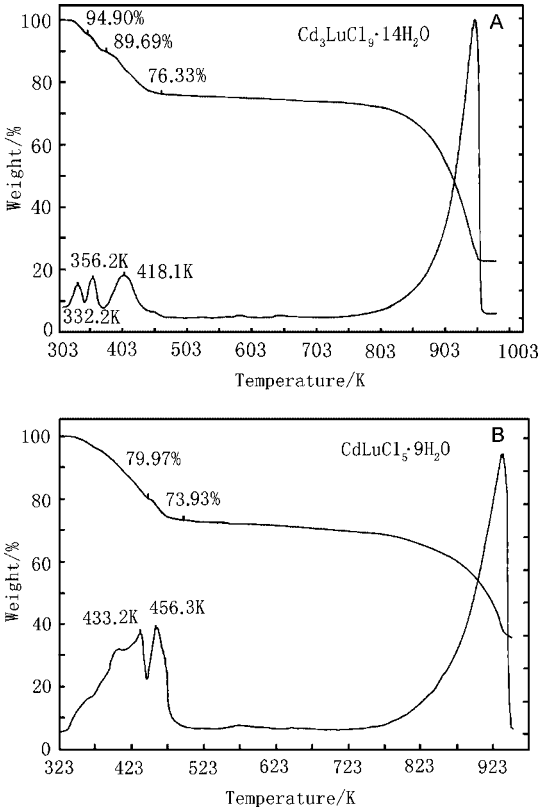

Figure 3. Thermogravimetric curves of $Cd_3LuCl_9 \cdot 14H_2O$ (A) and $CdLuCl_5 \cdot 9H_2O$ (B).

$\mu V \cdot mW^{-1}$. The reliability of the calorimeter was verified by measuring the enthalpy of solution of KCl(s) in deionized water. The average experimental value of $\Delta_{sol}H_m$(KCl) was determined to be (17.31 ± 0.06) $kJ \cdot mol^{-1}$ ($n = 6$), which is in excellent agreement with that of 17.234 $kJ \cdot mol^{-1}$ reported in the lierature.[13] The relative error between the literature and the measured value was 0.45 %. This shows that the device used for measuring the enthalpy of solution in this work is reliable.

Each calorimetric experiment was performed six times. The temperature of the calorimetric experiment was (298.15 ± 0.01) K. Water was put in the 15 cm^3 stainless steel sample cell and reference cell of the calorimeter. After thermal equilibration for at least 2 h, the solid sample was dissolved in deionized water. The thermal effect was then recorded automatically on a computer. The total time required for the complete dissolution was about 0.4 h. There were no solid residues observed after the dissolution in each calorimetric experiment.

Results and Discussion

$LuCl_3-CdCl_2-HCl$(7.47 mass %)$-H_2O$ Quaternary System at 298.15 K. The solubility data of the $LuCl_3-CdCl_2-HCl$(7.47 mass %)$-H_2O$ quaternary system and the central projection data on the trigonal basal face of $LuCl_3-CdCl_2-H_2O$ at 298.15 K are listed in Table 1 and plotted in Figure 1. It can be seen that the phase diagram of the $LuCl_3-CdCl_2-HCl-H_2O$ quaternary system consists of four solubility curves which correspond to the equilibrium solid phases $CdCl_2 \cdot H_2O$ (A), $Cd_3LuCl_9 \cdot 14H_2O$ (B) (3:1 type), $CdLuCl_5 \cdot 9H_2O$ (C) (1:1 type), and $LuCl_3 \cdot 6H_2O$ (D), respectively. The solid compounds $Cd_3LuCl_9 \cdot 14H_2O$ and $CdLuCl_5 \cdot 9H_2O$ are both congruently soluble in the medium of ~7.47 mass % HCl and are obtained easily. The two new compounds $Cd_3LuCl_9 \cdot 14H_2O$ and $CdLuCl_5 \cdot 9H_2O$ obtained from this system were analyzed by a titration method. Their compositions are 50.74 % $CdCl_2$ and 25.90 % $LuCl_3$ in $Cd_3LuCl_9 \cdot 14H_2O$ (theoretical, 50.76 % $CdCl_2$, 25.96 % $LuCl_3$) and 29.13 % $CdCl_2$ and 44.96 % $LuCl_3$ in $CdLuCl_5 \cdot 9H_2O$ (theoretical, 29.25 % $CdCl_2$, 44.89 % $LuCl_3$), respectively. This indicates that the formation of the solid compounds determined by the Schreinemakers method is reliable. It should be noted that both 3:1 type and 1:1 type compounds have not been reported up to now in the present system. Comparing the reported quaternary systems 1 to 8 with the present quaternary system, the phase behavior for Lu chloride differs from those of the light rare earth elements (La - Eu) and also differs from chlorides of the heavy rare earth elements such as Dy, Er, and Y. This indicates differences in behavior of the heavy rare earth elements in aqueous salt systems with $CdCl_2$.

Characterization of $Cd_3LuCl_9 \cdot 14H_2O$ and $CdLuCl_5 \cdot 9H_2O$. X-ray powder diffraction patterns of the $Cd_3LuCl_9 \cdot 14H_2O$ and $CdLuCl_5 \cdot 9H_2O$ compounds obtained are shown in Figure 2. The important X-ray data of the two compounds are d (nm) = 0.5847 (99), 0.5029 (24), 0.4576 (48), 0.3285 (31), 0.2783 (30), and 0.2586 (25) and d (nm) = 0.5690 (99), 0.4088 (16), 0.3108 (11), and 0.1929 (11). They are obviously different from the literature XRD data of the two starting salts: $CdCl_2$, d (nm) = 0.5850 (100), 0.3270 (70), 0.2648 (90), 0.2412 (30), 0.1826 (55), and 0.1922 (30), and $LuCl_3$, d (nm) = 0.2690 (100), 0.1935 (100), 0.5840 (80), 0.3300 (80), 0.1495 (80), and 0.1184 (80). This demonstrates that the two compounds are new.

TG-DTG data for the two compounds is presented in Figure 3. The curve for the compound $Cd_3LuCl_9 \cdot 14H_2O$, shows that there are three obvious mass-loss steps in the temperature range (318 to 452) K, and the total mass-loss value is in agreement with theoretical dehydration data. The same investigation was performed for $CdLuCl_5 \cdot 9H_2O$. One observes on the curve that, the two obvious mass-loss steps in the temperature range (333 to 484) K, the total mass-loss value is basically in accordance with theoretical dehydration data. On the basis of these results, we suggest that the dehydration equations for the two compounds are as follows

Table 2. Molar Enthalpies of Solution of $\Delta_{sol}H_m(1)$ and $\Delta_{sol}H_m(2)$ in Deionized Water at 298.15 K[a,b]

no.	m	$\Delta_{sol}H$	$\Delta_{sol}H_m(1)$	m	$\Delta_{sol}H$	$\Delta_{sol}H_m(2)$
	mg	mJ	$kJ \cdot mol^{-1}$	mg	mJ	$kJ \cdot mol^{-1}$
1	96.20	2582.90	29.09	55.68	−1400.61	−15.76
2	96.11	2578.81	29.07	55.91	−1401.64	−15.71
3	96.35	2567.39	28.87	55.47	−1357.82	−15.34
4	96.27	2540.62	28.59	55.27	−1329.75	−15.08
5	96.84	2539.71	28.41	55.19	−1325.86	−15.06
6	96.21	2552.43	28.74	55.56	−1410.32	−15.91
mean			28.79 ± 0.22			−15.48 ± 0.30

[a] In each experiment, 8.0 cm^3 of water was used. [b] Uncertainty is twice the standard deviation of the mean.

$$Cd_3LuCl_9 \cdot 14H_2O \xrightarrow[(318\ \text{to}\ 345)\,K]{-3H_2O} Cd_3LuCl_9 \cdot 1H_2O \xrightarrow[(345\ \text{to}\ 376)\,K]{-3H_2O}$$

$$Cd_3LuCl_9 \cdot 8H_2O \xrightarrow[(376\ \text{to}\ 452)\,K]{-8H_2O} Cd_3LuCl_9 \quad (1)$$

$$CdLuCl_5 \cdot 9H_2O \xrightarrow[(333\ \text{to}\ 445)\,K]{-7H_2O}$$

$$Cd_3LuCl_5 \cdot 5H_2O \xrightarrow[(445\ \text{to}\ 484)\,K]{-2H_2O} CdLuCl_5 \quad (2)$$

Enthalpies of Solution. The molar enthalpies of solution in water $\Delta_{sol}H_m(1)$ and $\Delta_{sol}H_m(2)$ of $Cd_3LuCl_9 \cdot 14H_2O$ and $CdLuCl_5 \cdot 9H_2O$ are (28.79 ± 0.22) $kJ \cdot mol^{-1}$ and (−15.48 ± 0.30) $kJ \cdot mol^{-1}$ at 298.15 K, respectively. The values are listed in Table 2, in which m is the mass of the sample and $\Delta_{sol}H_m$ is the molar enthalpy of solution of the sample. The uncertainty is estimated as twice the standard deviation of the mean,[10] namely

$$\delta = 2\sqrt{\sum (x_i - \bar{x})^2/n(n-1)}$$

in which n is the number of experimental measurements ($n = 6$); x_i is the experimental value of each repeated measurement; and $\bar{x}$ is the mean value.

Standard Molar Enthalpies of Formation. The molar enthalpies of formation of $Cd_3LuCl_9 \cdot 14H_2O$ and $CdLuCl_5 \cdot 9H_2O$ can be calculated as shown in following equations

$$\Delta_f H_m^{\Theta}(Cd_3LuCl_9 \cdot 14H_2O)(s) = 3\Delta_f H_m^{\Theta}(Cd^{2+})(aq) + \Delta_f H_m^{\Theta}(Lu^{3+})(aq) + 9\Delta_f H_m^{\Theta}(Cl^-)(aq) + 14\Delta_f H_m^{\Theta}(H_2O)(l) - \Delta_{sol} H_m^{\Theta}(Cd_3LuCl_9 \cdot 14H_2O)(s)$$

$$\Delta_f H_m^{\Theta}(CdLuCl_5 \cdot 9H_2O)(s) = \Delta_f H_m^{\Theta}(Cd^{2+})(aq) + \Delta_f H_m^{\Theta}(Lu^{3+})(aq) + 5\Delta_f H_m^{\Theta}(Cl^-)(aq) + 9\Delta_f H_m^{\Theta}(H_2O)(l) - \Delta_{sol} H_m^{\Theta}(CdLuCl_5 \cdot 9H_2O)(s)$$

The standard molar enthalpies of formation of Cd^{2+}, Cl^-, and H_2O were taken from the CODATA Key Values,[14] namely, (−75.90 ± 0.20) $kJ \cdot mol^{-1}$, (−167.16 ± 0.10) $kJ \cdot mol^{-1}$, and (−285.83 ± 0.042) $kJ \cdot mol^{-1}$ for Cd^{2+}, Cl^-, and H_2O, respectively. Lu^{3+} was taken from the NBS table,[15] (−665.00 ± 0.10) $kJ \cdot mol^{-1}$. Using these schemes and data, the standard molar enthalpies of formation of $Cd_3LuCl_9 \cdot 14H_2O$ and $CdLuCl_5 \cdot 9H_2O$ were calculated to be (−6427.5 ± 1.3) $kJ \cdot mol^{-1}$ and (−4133.7 ± 0.7) $kJ \cdot mol^{-1}$, respectively.

Conclusion

The solubilities of the quaternary system $LuCl_3-CdCl_2-HCl$(7.47 mass %)$-H_2O$ at 298.15 K were measured, and the corresponding phase diagram was prepared to search for new compounds and obtain the equilibrium data for cadmium chloride/rare earth metal chloride in aqueous solution. The compositions of the solid phases were determined by Schrienemakers method and confirmed by chemical analysis. Both the compounds $Cd_3LuCl_9 \cdot 14H_2O$ (3:1 type) and $CdLuCl_5 \cdot 9H_2O$ (1:1 type) are congruently soluble in the quaternary system. Two kinds of new compounds, 3:1 type and 1:1 type, were found to exist in the present system and were not found in the earlier reported systems $RECl_3-CdCl_2-HCl-H_2O$ (RE = La, Ce, Pr, Nd, Eu, Dy, Y, Er). Through measuring the standard molar enthalpy of solution, the standard molar enthalpies of formation of the two new compounds were calculated.

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