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Solid−Liquid Equilibria in the Ternary Systems NaCl−SrCl$_2$−H$_2$O and KCl−SrCl$_2$−H$_2$O at 348 K

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ABSTRACT: Solid−liquid equilibria in the ternary systems NaCl−SrCl$_2$−H$_2$O and KCl−SrCl$_2$−H$_2$O at 348 K were measured by the isothermal solution saturation method. The composition of the equilibrium solid phase, solubilities of salts, and densities of saturated solution in the two systems were determined. Phase diagrams of ternary systems were plotted according to experimental data. They both contained one invariant point, two univariant curves, and two regions of crystallization. Respectively, univariant curves of the corresponding equilibrium solid phase were sodium chloride (NaCl) and strontium chloride dihydrate (SrCl$_2$·2H$_2$O) in the ternary system NaCl−SrCl$_2$−H$_2$O, and potassium chloride

(KCl) and strontium chloride dihydrate (SrCl$_2$·2H$_2$O) in the ternary system KCl−SrCl$_2$−H$_2$O. The two ternary systems belong to the simple type, and neither double salts nor solid solution was found. The density transformation rules were simply discussed. Simultaneously, the solubilities and density data were compared with the experimental data of previous researchers.
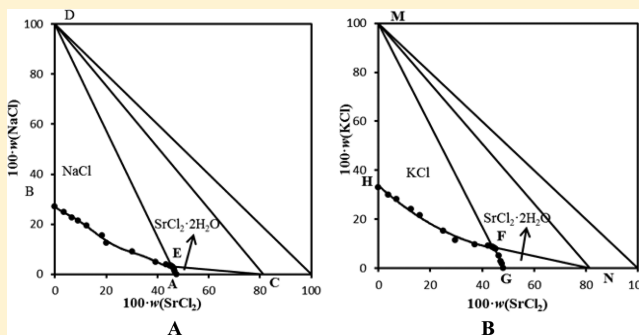
1. INTRODUCTION

Underground brine is widely distributed in the Sichuan basin of China, and the development has a long history. The underground brines rich in resources are a distinct advantage of liquid mineral resources in Sichuan.[1] To exploit and utilize these valuable brine resources, their relevant thermodynamic data, particularly the solubilities of salts in the brines at different temperatures, are necessary. By now, a series of works on phase equilibria aiming at the brines have been done by our group, such as the quinary systems Li$^+$,Na$^+$,K$^+$//CO$_3^{2−}$,B$_4$O$_7^{2−}$−H$_2$O at 288 K,[2] the quinary systems K$^+$//Cl$^−$,Br$^−$,B$_4$O$_7^{2−}$,SO$_4^{2−}$−H$_2$O at 323 K and 348 K,[3] the quaternary systems K$^+$//Cl$^−$,Br$^−$,B$_4$O$_7^{2−}$−H$_2$O and K$^+$//Cl$^−$,Br$^−$,SO$_4^{2−}$−H$_2$O at 348 K.[4]

The brine of the Sichuan basin contains a high amount of potassium and boron.[5,6] Our research group has also carried out many measurements of phase equilibria containing potassium, such as the quinary systems Na$^+$,K$^+$//Cl$^−$, SO$_4^{2−}$, B$_4$O$_7^{2−}$−H$_2$O,[7] the quaternary system Na$^+$, K$^+$// Br$^−$, SO$_4^{2−}$−H$_2$O at 323 K,[8] and the quinary system Na$^+$,Mg^{2+}, K$^+$//SO$_4^{2−}$,B$_4$O$_7^{2−}$−H$_2$O at 288 K.[9] Beside NaCl, the brines also include high contents of useful elements such as Sr, I and Br.[10]

Strontium (Sr) and its compounds play very important roles in many industrial fields.[11] At present, the main Sr resources in China are solid ore deposits. Also, the underground brines in the mineralization belts with associated Sr in the Sichuan basin are rich in this element.[12] So it is necessary to study the phase equilibria and phase diagrams of a Sr-rich underground brine system. The ternary systems NaCl−SrCl$_2$−H$_2$O and KCl−SrCl$_2$−H$_2$O are two important and basic strontium subsystems of the underground brine in the Western Sichuan Basin.

Many measurements of phase equilibria containing strontium have been performed, for example, measurements of the ternary systems Na$^+$,Sr^{2+}//Cl$^−$−H$_2$O, K$^+$,Sr^{2+}//Cl$^−$−H$_2$O and Ca^{2+},Sr^{2+}//Cl$^−$−H$_2$O at 298 K,[13−15] the quaternary system Na$^+$,K$^+$,Sr^{2+}//Cl$^−$−H$_2$O at 291 K to 373 K,[16] the quinary systems Na$^+$,K$^+$,Sr^{2+},Ca^{2+}//Cl$^−$−H$_2$O at 291 K to 387 K,[17] the polycomponent aqueous systems Na$^+$,K$^+$,Sr^{2+},Ca^{2+},Mg^{2+}// Cl$^−$−H$_2$O between 291 K and 366 K,[18] and the ternary systems Ca^{2+},Sr^{2+}//Cl$^−$−H$_2$O between 291 K and 386 K have been published,[19] but no reports have been found to describe the phase equilibria of NaCl−SrCl$_2$−H$_2$O and KCl−SrCl$_2$− H$_2$O at 348 K. Therefore, the phase equilibria of these two ternary systems were studied in detail in this paper.

2. EXPERIMENTS

2.1. Reagents and Instruments. Deionized water (electrical conductivity less than 1.2·10$^{−4}$ S·m$^{−1}$) was obtained from a Millipore water system, and the water was used to prepare the experimental solutions. All the chemicals used in this work were analytical purity grade: strontium chloride hexahydrate (SrCl$_2$·6H$_2$O; ≥ 99.0 wt %), sodium chloride (NaCl; ≥ 99.5 wt %), and potassium chloride (KCl; ≥ 99.5 wt %).

A standard analytical balance of 110 g capacity and 0.0001 g resolution (AL104), made by the Mettler Toledo Instruments was used for the determination of the solution density.

An SHA-GW type oil bath thermostated vibrator made by Jintan Guowang instrument plant with an uncertainty of 0.1 K was used for the equilibrium experiments.

2.2. Experimental Method. The phase equilibrium of the ternary systems was determined at 348 K by the isothermal solution saturation method. The system points for the ternary systems were obtained by adding the second component gradually on the basis of the first salt saturation points at 348 K. According to the literature, the prepared two kinds of salt were dissolved into 25 mL of water in a sealed glass bottle. Then the bottles were placed into the oil bath vibrator to make the system uniform and reach equilibrium gradually. Generally speaking, the equilibrium states can be established in 15 days at 348 K.[20] The supernatant fluids were taken out periodically for chemical analysis. When the the concentration of the solutions did not change any more, it meant that equilibrium was established in this system. The sealed glass bottles were allowed to settle for one week in the oil bath, which was controlled at 348 ± 0.1 K. After the liquid clarified, the supernatant fluid and wet solid phase were removed for physicochemical analysis. The compositions of the solutions were analyzed quantitatively by chemical analysis, and the solid phases after natural drying were identified by X-ray diffraction (XRD; Siemens D500 X-ray diffract meter). The densities were measured using the pycnometer method by a density bottle (with a precision of 0.0002 g·cm^{-3}).

2.3. Analytical Methods.[21] The potassium ion concentration (K^+) was determined using sodium tetraphenylborate−hexadecyl trim ethylammonium bromide titration (uncertainty, $U_r(w(K^+)) = 0.0054$). The chloride ion concentration (Cl^-) was determined by Mohr's method with a silver nitrate standard solution (uncertainty, $U_r(w(Cl^-)) = 0.0032$). The strontium ion concentration (Sr^{2+}) was determined by ethylene diamine tetraacetic acid solution, K−B indicator (acid chrome blue K/naphtha green B = 1:2) was employed as indicator (uncertainty, $U_r(w(Sr^{2+})) = 0.0078$).[15] Sodium ion concentration (Na^+) was evaluated according to the ion charge balance.

3. RESULTS AND DISCUSSION

3.1. NaCl−SrCl$_2$−H$_2$O System at 348 K. The determined solubilities and densities for the ternary system NaCl−SrCl$_2$−H$_2$O at 348 K were measured and presented in Table 1. The ion concentration values were expressed in mass fraction $w(a)$, and ρ is the density value. On the basis of the experimental data in Table 1, the equilibrium phase diagram of this system at 348 K is shown in Figure 1. The phase diagram of the system in Figure 1 consists of two crystallization fields corresponding to sodium chloride (NaCl) and strontium chloride dihydrate (SrCl$_2$·2H$_2$O). In this system, there is one invariant point, noted as E. Point A was the single salt saturation of SrCl$_2$·2H$_2$O and the Point B is the single salt saturation of NaCl accordingly. Point C was the solid phase composition of SrCl$_2$·2H$_2$O and Point D was the solid phase composition of NaCl. Point E was saturated with two salts: NaCl and SrCl$_2$·2H$_2$O, and the mass fraction composition of the corresponding liquid phase was $w(SrCl_2) = 0.4554$, $w(NaCl) = 0.0312$. There were two univariant curves corresponding to curves AE and BE. The crystallization field of salt SrCl$_2$·2H$_2$O (AEC) was the smallest, while the crystallization field of salt NaCl (BED) was the largest. The area (DEC) was the crystallization area of NaCl and SrCl$_2$·2H$_2$O.

On the basis of data listed in Table 1, the relationship between density and the mass fraction of strontium chloride

Table 1. Solubilities and Densities of Solution in the Ternary System NaCl−SrCl$_2$−H$_2$O at 348 K and 0.1 MPa^a

| no. | composition of solution $w(a)\cdot100$ | | solid phase | density ρ/g·cm^{-3} |
	$w(SrCl_2)$	$w(NaCl)$		
1(A)	47.75	0.00	SrCl$_2$·2H$_2$O	1.6205
2	46.76	1.22	SrCl$_2$·2H$_2$O	1.6171
3	46.60	1.95	SrCl$_2$·2H$_2$O	1.6126
4	46.49	2.17	SrCl$_2$·2H$_2$O	1.6087
5	46.03	2.96	SrCl$_2$·2H$_2$O	1.5998
6(E)	45.54	3.12	SrCl$_2$·2H$_2$O + NaCl	1.5912
7	44.86	3.30	NaCl	1.5893
8	44.37	3.33	NaCl	1.5631
9	44.02	3.36	NaCl	1.5675
10	43.30	3.80	NaCl	1.5482
11	39.27	4.75	NaCl	1.5250
12	30.14	9.03	NaCl	1.4437
13	20.11	12.40	NaCl	1.3712
14	18.34	15.46	NaCl	1.3398
15	12.46	19.32	NaCl	1.2944
16	9.06	21.48	NaCl	1.2432
17	6.59	22.64	NaCl	1.2308
18	3.46	24.96	NaCl	1.1935
19(B)	0.00	27.45	NaCl	1.1830

^{a}$w(a)$ is the mass fraction of component a. Relative expanded uncertainties (level of confidence =0.95): $U_r(T) = 0.1$ K; $U_r(w(Cl^-))$ = 0.0032; $U_r(w(Sr^{2+})) = 0.0078$; $U_r(\rho) = 0.0002$ g·cm^{-3}.
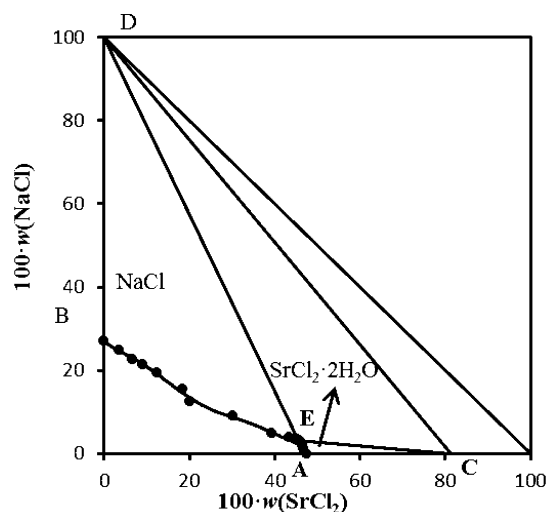

Figure 1. Equilibrium phase diagram of the ternary system NaCl−SrCl$_2$−H$_2$O at 348 K and 0.1 MPa.

$w(SrCl_2)$ and $w(NaCl)$ are shown in Figure 2, $w(SrCl_2)$ as X coordinate axis, $w(NaCl)$ as Y coordinate axis and density value as Z coordinate axis. In this system, the concentration of SrCl$_2$ was the main factor affecting the solution density. According to the experimental data, the density value had changed along with the content change of NaCl and SrCl$_2$. Obviously, the density value increases with increasing content of SrCl$_2$ and decreasing content of NaCl as shown in Figure 2.

Solid phase composition was determined using X-ray diffraction. Figure 3 is an X-ray diffraction photograph of a date point on univariant curve AE of the ternary system NaCl−SrCl$_2$−H$_2$O at 348 K. Figure 4 is the X-ray diffraction photograph of the equilibrium solids NaCl and SrCl$_2$·2H$_2$O
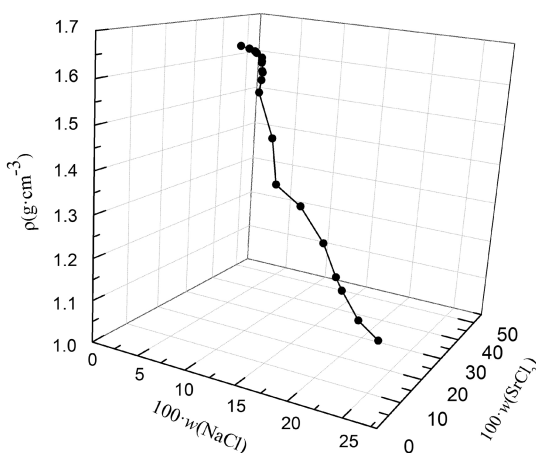
Figure 2. Density value vs composition of the ternary system NaCl–SrCl$_2$–H$_2$O at 348 K and 0.1 MPa.
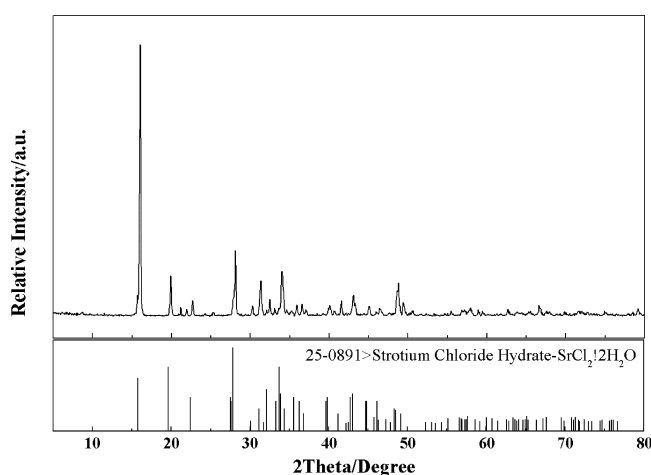

Figure 3. X-ray diffraction photograph of a date point of the ternary system NaCl–SrCl$_2$–H$_2$O at 348 K.

for the invariant point of the ternary system NaCl–SrCl$_2$–H$_2$O at 348 K.

3.2. KCl–SrCl$_2$–H$_2$O System at 348 K. The experimental results of solubilities and densities for the ternary system KCl–
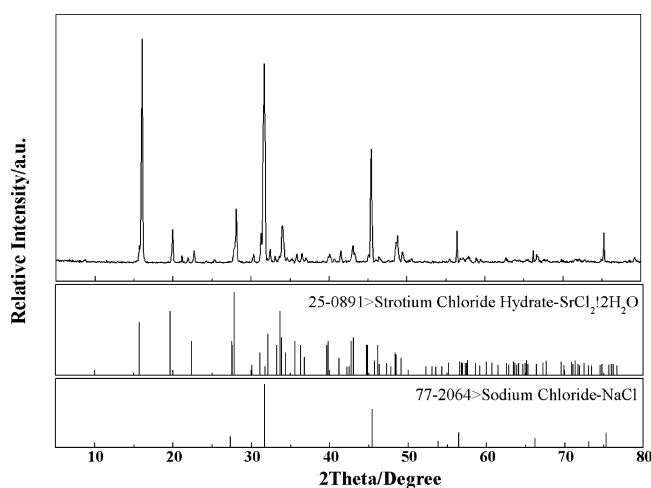

Figure 4. X-ray diffraction photograph of the invariant point of the ternary system NaCl–SrCl$_2$–H$_2$O at 348 K.

SrCl$_2$–H$_2$O at 348 K were measured and presented in Table 2. With the experimental data, the equilibrium phase diagram of

Table 2. Solubilities and Densities of Solution in the Ternary System KCl–SrCl$_2$–H$_2$O at 348 K and 0.1 MPa^a

	composition of solution $w(b) \cdot 100$			density
no.	$w(SrCl_2)$	$w(KCl)$	solid phase	ρ/g·cm^{-3}
1(G)	47.75	0.00	SrCl$_2$·2H$_2$O	1.6205
2	47.37	1.81	SrCl$_2$·2H$_2$O	1.6264
3	47.02	2.75	SrCl$_2$·2H$_2$O	1.6331
4	46.42	5.09	SrCl$_2$·2H$_2$O	1.6442
5	45.25	7.88	SrCl$_2$·2H$_2$O	1.6495
6	44.88	8.19	SrCl$_2$·2H$_2$O	1.6594
7	44.58	8.39	SrCl$_2$·2H$_2$O	1.6654
8(F)	44.04	8.57	SrCl$_2$·2H$_2$O + KCl	1.6694
9	42.18	9.14	KCl	1.6325
10	37.07	9.66	KCl	1.5689
11	29.51	11.40	KCl	1.4705
12	24.94	15.14	KCl	1.4254
13	16.07	21.43	KCl	1.3296
14	12.65	24.12	KCl	1.3080
15	6.95	28.09	KCl	1.2607
16	3.83	29.93	KCl	1.2357
17(H)	0.00	33.20	KCl	1.2040

^{a}$w(b)$ is the mass fraction of component b. Relative expanded uncertainties (level of confidence = 0.95): $U_r(T) = 0.1$ K; $U_r(w(Cl^-)) = 0.0032$; $U_r(w(K^+)) = 0.0054$; $U_r(w(Sr^{2+})) = 0.0078$; $U_r(\rho) = 0.0002$ g·cm^{-3}.
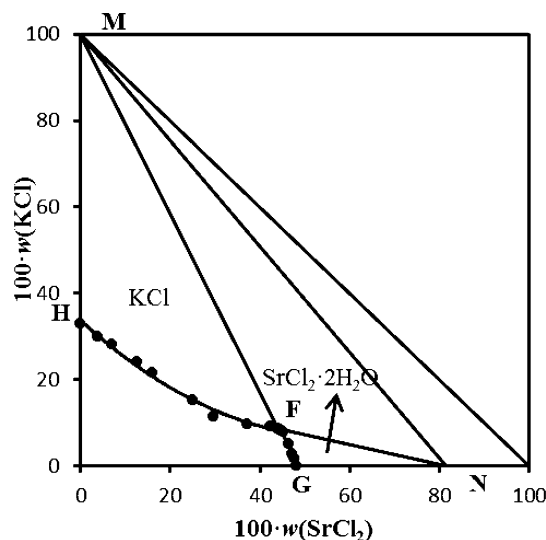

Figure 5. Equilibrium phase diagram of the ternary system KCl–SrCl$_2$–H$_2$O at 348 K and 0.1 MPa.

this system at 348 K was plotted in Figure 5. The phase diagrams of the system in Figure 5 were two crystallization fields, one eutectic point, and two univariant curves. No double salt or solid solution was found in this system at 348 K. The two crystallization fields corresponding to the two single salts KCl and SrCl$_2$·2H$_2$O.

The crystallization field of salt SrCl$_2$·2H$_2$O (FGN) was the smallest, while the crystallization field of salt KCl (FHM) was

the largest. The area (FMN) was the crystallization area of KCl and $SrCl_2 \cdot 2H_2O$. Point G was the single salt saturation of $SrCl_2$ and the point H was the single salt saturation of KCl accordingly. Point N was the solid phase composition of $SrCl_2 \cdot 2H_2O$ and point M was the solid phase composition of KCl. Point F was the eutectic point, and it was saturated with two salts: KCl and $SrCl_2 \cdot 2H_2O$. The mass fraction composition of the corresponding liquid phase was w ($SrCl_2$) = 0.4404, w (KCl) = 0.0857. The two univariant curves corresponded to curves HF and FG in this system.

The density data are shown in Figure 6 on the basis of data listed in Table 2. Figure 6 shows $w(SrCl_2)$ as the X coordinate
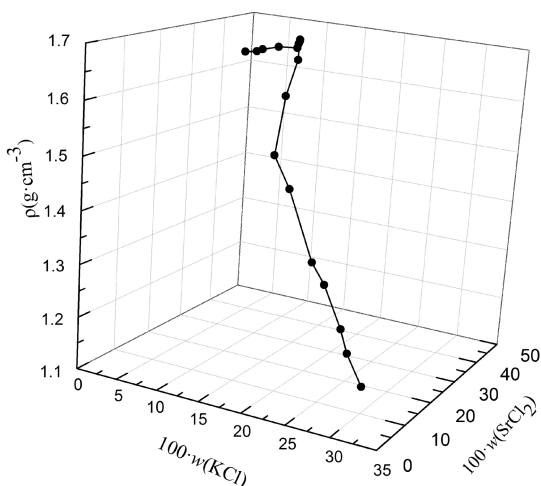

Figure 6. Density value vs composition of the ternary system KCl–$SrCl_2$–H_2O at 348 K and 0.1 MPa.

axis, w(KCl) as the Y coordinate axis, and the density value as the Z coordinate axis. In this ternary system at 348 K, the solubility of the salt $SrCl_2$ was greater than that of the salt KCl. Obviously, the density value increases with increasing content of $SrCl_2$. The density value first increases in the $w(SrCl_2)$ range of 0 % to 44.04 %. At the eutectic point F, the density reaches the maximum value that is 1.6694 $g \cdot cm^{-3}$. Then, there is a small reduction in the $w(SrCl_2)$ range of 44.04 % to 47.75 %.

The solid phase composition had been shown by the X-ray diffraction. Figure 7 is the X-ray diffraction photograph of a
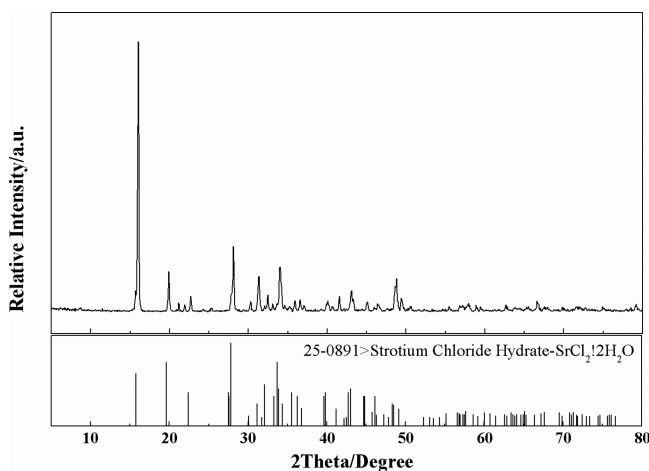

Figure 7. X-ray diffraction photograph of a date point of the ternary system KCl–$SrCl_2$–H_2O at 348 K.

date point on univariant curves FG of the ternary system KCl–$SrCl_2$–H_2O at 348 K. Figure 8 is the X-ray diffraction photograph of the equilibrium solids KCl and $SrCl_2 \cdot 2H_2O$ for the invariant point of the ternary system KCl–$SrCl_2$–H_2O at 348 K.
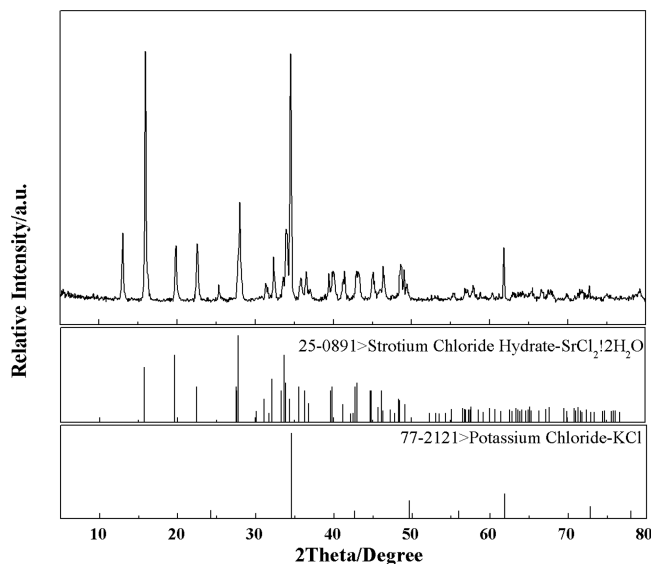

Figure 8. X-ray diffraction photograph of the invariant point of the ternary system KCl–$SrCl_2$–H_2O at 348 K.

3.3. Comparison of the Ternary Systems NaCl–$SrCl_2$–H_2O and KCl–$SrCl_2$–H_2O at 348 K. Comparing Figure 1 and Figure 5, we found that the ternary systems NaCl–$SrCl_2$–H_2O and KCl–$SrCl_2$–H_2O both contained one invariant point, two univariant curves, and two regions of crystallization. The crystallization fields of salt $SrCl_2 \cdot 2H_2O$ were both smaller than the crystallization fields of salt NaCl(KCl); therefore the $SrCl_2$ had larger solubility than NaCl(KCl), in these ternary systems, because $SrCl_2$ had a stronger salting-out effect than NaCl(KCl).

3.4. Discussion of Strontium Chloride Dihydrate in the System. From the phase diagram and solid phase identification it can be seen that $SrCl_2 \cdot 6H_2O$ had lost four crystallization water molecules to form $SrCl_2 \cdot 2H_2O$. According to ref 22, we can find the number crystallization changes of $SrCl_2$ with water with the temperature. The research result from ref 22 suggests that $SrCl_2 \cdot 6H_2O$ dehydration occurs at 292.4 K, and $SrCl_2 \cdot 2H_2O$ is fully formed at 292.9 K. As temperatures continue to rise to 354 K $SrCl_2 \cdot H_2O$ is generated; therefore, the crystalline region solid phase is $SrCl_2 \cdot 2H_2O$ at 348 K. From Figures 3, 4, 7, and 8 for solid phase XRD testing results in this work we also found that the number of crystal water was consistent with the literature results at the corresponding temperatures, showing that the results in this work were credible.

3.5. Comparison with the Experimental Data of Previous Researchers. In ref 16 the results for aqueous systems containing Na^+, K^+, Sr^{2+}, and Cl^- were reported.[16] The solubilities in the two systems were determined at 291 K, 333 K, and 373 K. The comparison between our experimental data and the data from that paper is shown in Figure 9 and Figure 10, As the temperature rises, the solubility curves of two systems both move into the direction of the total salinity increasing, and the liquid salts concentration increases. The solubility curves conformed to the trend at 348 K.
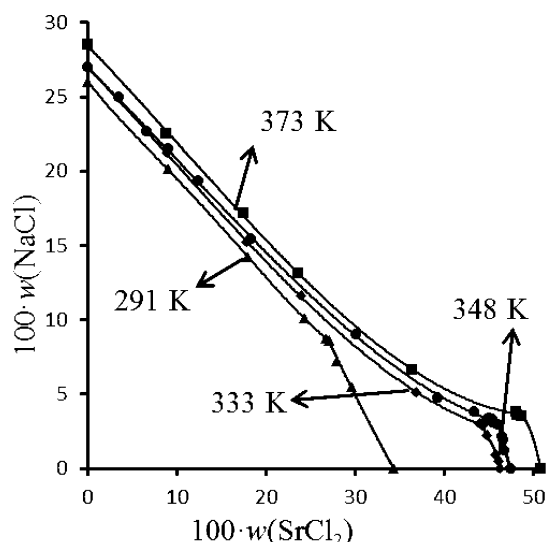
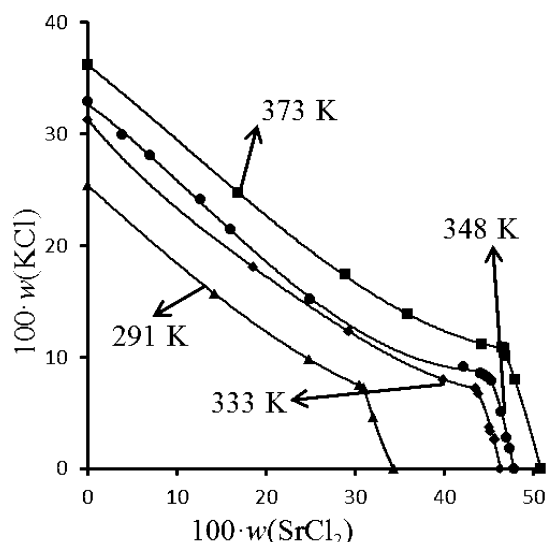**Figure 9.** Solubilities data comparison of the ternary system NaCl–SrCl₂–H₂O at 291 K, 333 K, 348 K, and 373 K.[16]

Figure 10. Solubilities data comparison of the ternary system KCl–SrCl₂–H₂O at 291 K, 333 K, 348 K, and 373 K.[16]

Simultaneously, the data of invariant points of two systems had been compared in Table 3. The results indicated that the solubility of the salts increased from 333 K to 373 K. In

Table 3. Data of Invariant Points Comparison of Two Ternary Systems at 333 K, 343 K, 348 K, 353 K, and 373 K[16] [a]

| | NaCl–SrCl₂–H₂O System | | KCl–SrCl₂–H₂O System | |
| | composition of solution $w(c)\cdot100$ | | composition of solution $w(c)\cdot100$ | |
temp	$w(SrCl_2)$	$w(NaCl)$	$w(SrCl_2)$	$w(KCl)$
333 K	44.3	2.9	43.5	7.2
343 K	44.9	3.1	43.9	8.2
348 K	45.5	3.1	44.0	8.6
353 K	45.9	3.3	44.5	8.9
373 K	48.2	3.6	46.7	10.8

[a] $w(c)$ is the mass fraction of component c.

addition, according to the density data from refs 13 and 14, our experimental data conformed to the trend at 348 K, showing the data and conclusion of this article were credible.

4. CONCLUSIONS

Phase equilibria in the ternary systems NaCl–SrCl₂–H₂O and KCl–SrCl₂–H₂O were investigated at 348 K using an isothermal solution saturation method. The phase diagrams and density diagrams of the two systems were plotted according to the experimental data. The results showed that these two ternary systems were a type of simple common-saturation, and they both had one invariant point, two univariant curves, and two regions of crystallizations. The two crystallization regions of the ternary system NaCl–SrCl₂–H₂O at 348 K corresponded to SrCl₂·2H₂O and NaCl. The two fields of crystallization were SrCl₂·2H₂O and KCl in the ternary system KCl–SrCl₂–H₂O at 348 K. In these two ternary systems the SrCl₂·6H₂O had lost four crystallization water molecules to form SrCl₂·2H₂O at 348 K.

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Notes
The authors declare no competing financial interest.

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