Studies of Phase Equilibria in Ternary KBr–$SrBr_2$–H_2O and NaBr–$SrBr_2$–H_2O Systems at 308 K

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The phase equilibria of the KBr–$SrBr_2$–H_2O and NaBr–$SrBr_2$–H_2O ternary systems at 308 K were measured using the isothermal dissolution equilibrium method. The compositions of the liquid and solid phases were expressed as the mass fraction, and the phase diagrams constructed from the experimental data show that both ternary systems are simple co-saturated types with only one invariant point, two univariant curves, and two crystallization regions. Neither of the two ternary systems forms a double-salt or solid solution. The solid-phase crystallization regions of the KBr–$SrBr_2$–H_2O ternary system correspond to KBr and $SrBr_2 \cdot 6H_2O$, and the invariant point of the NaBr–$SrBr_2$–H_2O ternary system is saturated with $NaBr \cdot 2H_2O$ and $SrBr_2 \cdot 6H_2O$. From the phase diagrams, the salting-out effect of $SrBr_2$ on KBr is clear, which indicates that the solubility of KBr is lower and it is easier to separate KBr from solution in the ternary KBr–$SrBr_2$–H_2O system at 308 K.

Introduction

The Sichuan Basin of China is a giant free-flowing basin with abundant underground brine resources and numerous production layers. The Sichuan Basin is rich in brine resources with an area of over 2×10^5 km (Lin 2001; Lin *et al.*, 2002) and is rich in lithium, potassium, rubidium, magnesium, boron, bromine, strontium, etc. The brines are of good quality with a high mining value. The content of many useful components in the brines exceed the grades of industrial exploitation. In China, strontium ores are abundant, especially liquid strontium ore. These ores are easily exploited and cause little pollution and damage to the environment (Lin, 2006). Therefore, the strontium-rich brines in the Sichuan Basin have very high value for development and utilization. Most of the underground brines accompanying the strontium metallogenic belt are present in different brine layers at depths of 50–3000 m underground in the Sichuan Basin. From calculation of the average annual temperature of the basin at 15°C and the geothermal gradient of the basin at 2.3°C/100 m, the temperature of the underground brines ranges from 289.3 to 357.2 K. In the actual mining process, as the brine is pumped out from the underground reservoir, the pressure and temperature both decrease; therefore, the temperature of pumped-out underground brines is often lower than that of underground brine (Lin *et al.*, 2004). Therefore, study of the phase equilibria of underground brines in the Sichuan Basin is necessary and can be carried out at 308 K.

The strontium-rich and potassium-rich brines in the Sichuan Basin are of excellent quality, making their exploitation promising for the national economy. Therefore, study of the phase equilibria of brine-salt systems in underground brines is indispensable, where the brines in the west of the Sichuan Basin are rich in strontium and potassium (Lin and Cao, 1998; Zhu, 1998). Moreover, such studies can provide theoretical guidance for the exploitation of salt lakes in China, and will promote the development of the potash fertilizer industry and brine chemical industry in China, and bring great social and economic benefits by further enhancing the recovery and utilization of useful components of underground brines in the Sichuan Basin. In order to realize this aim, it is necessary to grasp the basic rules of brine salting-out and mineralizationto to provide a scientific and effective guide for the production process.

At present, the phase equilibria of water-salt systems containing strontium are mostly studied in chloride systems. As early as the 1950s, Assarsson and coworkers in Sweden carried out a series of studies on the phase equilibria of water-salt systems containing strontium, all of which were based on systems (Assarsson, 1953; Assarsson and Balder, 1954a, 1954b, 1955) containing strontium chloride, specifically subsystems of the Na–K–Ca–Mg–Sr–Cl–H_2O system. Comprehensive studies of related phase equilibria have been undertaken by many scholars, including the phase equilibria of ternary systems such as NaCl–$SrCl_2$–H_2O at 288 K and KCl–$SrCl_2$–H_2O, LiCl–$SrCl_2$–H_2O, and $CaCl_2$–$SrCl_2$–H_2O at 298 K (Bi *et al.*, 2010; Shi *et al.*, 2010; Guo *et al.*, 2012; Li

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et al., 2016). Based on the characteristics of strontium-rich brines in the Sichuan Basin, in recent years, our research group has focused on the phase equilibria of the ternary KCl–$SrCl_2$–H_2O system at 323 and 348 K, as well as that of $MgCl_2$–$SrCl_2$–H_2O at 348 K (Li *et al.*, 2015a, 2015b; Zhang *et al.*, 2015). Several bromine-containing systems have also been evaluated. For example, the phase equilibria of the ternary $MgBr_2$–$SrBr_2$–H_2O system has been determined at 298 and 323 K (Liu *et al.*, 2018). The phase equilibria of the quaternary NaBr–$MgBr_2$–$SrBr_2$–H_2O and KBr–$MgBr_2$–$SrBr_2$–H_2O systems at 323 K were also reported (Liu *et al.*, 2017), and the solubility data for the quaternary NaBr–KBr–$MgBr_2$–H_2O and NaBr–KBr–$CaBr_2$–H_2O systems at 348 K were determined (Hu *et al.*, 2015a). The phase equilibria of the NaBr–KBr–$SrBr_2$–H_2O quaternary system at 323 and 348 K have been comprehensively documented (Hu *et al.*, 2015b; Cui *et al.*, 2017). The phase equilibria of the KBr–$SrBr_2$–H_2O and NaBr–$SrBr_2$–H_2O ternary systems at 288, 323, and 348 K, which are closely related to the system studied herein, have been published (Hu *et al.*, 2015b; Cui, 2017; Cui *et al.*, 2017; Li *et al.*, 2018), but the data at 308 K have not been reported. In this work, experimental studies of two ternary systems are carried out and phase diagrams of the systems are constructed, thereby providing an important supplement to the compendium on the multi-temperature and multi-phase equilibria of brines and providing a guide for the extraction of strontium and potassium salts.

1. Experimental

1.1 Reagents and instruments

All chemicals used in the experiment were of analytical purity; the specific details are presented in **Table 1**. Deionized water was used for the experiments; the pH was 6.6, and the conductivity was not less than $1\times10^{-5}\,S\cdot m^{-1}$. The oven used in the experiment was produced in Shanghai. An analytical balance was used to weigh the required chemical reagents. The stable phase equilibria of the ternary systems were evaluated using a constant temperature water-bath oscillator; the temperature of the systems was stable at 308±0.1 K. The water-bath oscillator was also used to accelerate equilibration. The solid phases were identified via X-ray diffraction. The instruments used in the experiment are listed in **Table 2**.

1.2 Experimental methods

For the two ternary systems studied in this work, the isothermal dissolution equilibrium method was adopted in the experiment. Firstly, 50 mL glass bottles were equilibrated to constant temperature; the second salt was added successively from the invariant point of the first salt, where the concentration of the second salt was varied at a certain interval. Deionized water was then added to the mixtures. The bottles containing the mixture were packed into a constant temperature water-bath oscillator and the temperature was controlled at 308 K (±0.1 K) to achieve equilibrium. In the early stage, oscillation was required to accelerate equilibration of the system. Before sampling and analysis, the oscillator was turned off and the sample bottles were kept completely stationary for several days, allowing the liquid particles to settle completely. Thereafter, the composition of the liquid phase of the upper layer was analyzed regularly. When the liquid phase composition consistently remained constant, the system was considered to be in equilibrium. After confirming equilibration of the system, the supernatant was taken for analysis and determination. A proper amount of the equilibrium liquid phase was pipetted into a volumetric flask and the mass was recorded. The liquid phase was subjected to chemical analysis for determination of the liquid phase point; the lower solid phases were subjected to chemical analysis and X-ray diffraction identification. A spoon was used to take an appropriate amount of the solid phase with a little of the equilibrium liquid phase, termed the wet solid phase, which was placed into a volumetric flask, then weighed, and the mass was recorded. After dissolution, the mass fraction of the wet slag was determined by chemical

Table 1 Chemical reagents

Chemical name	CAS	Purity	Source	Analysis method
Potassium bromide (KBr)	7758-02-3	≥99%	Chengdu Kelong Chemical Reagent Manufactory, China	Chemical titration
Sodium bromide (NaBr)	7647-15-6	≥99%	Chengdu Kelong Chemical Reagent Manufactory, China	Chemical analysis
Strontium bromide Hexahydrate ($SrBr_2\cdot6H_2O$)	7789-53-9	≥99%	Aladdin Industrial Corporation	Chemical titration

Table 2 Experimental instruments

Instrument name	Type and accuracy	Source	Area
Ultrapure Water device	UPT-II-20T	Youpu Chaochun Technology Co., Ltd.	Sichuan
X-ray diffraction	DX-2700	Danfangyuan Instrument Co., Ltd.	Shenzhen
Analytical balance	AL104, 0.0001 g	Mettler Toledo Instruments Co., Ltd.	Shanghai
Water bath oscillator	HZS-HA, ±0.1 K	Donglian Electronic Technology Development Co., Ltd.	Harbin
Oven	DHG-9076A	Jinghong Experimental Equipment Co., Ltd.	Shanghai

analysis method and plotted in the phase diagram, that is, as the wet slag point.

1.3 Analytical methods

In this work, chemical analysis of the potassium, strontium, and bromide ions in the systems was carried out. Firstly, the silver nitrate method was used to measure the total content of bromine ions in the solution, and potassium chromate was used as an indicator in near neutral solution (uncertainty: 0.3%). Thereafter, the content of potassium ions in solution was investigated by the sodium tetraphenylborate-quaternary ammonium salt back titration method (uncertainty: 0.5%), and the strontium ion content in the liquid phase was determined by the EDTA volumetric method with chromium black T as the indicator (uncertainty: 0.5%). Finally, the content of sodium ions was determined by the ion balance subtraction method. The wet solid phase method and X-ray diffraction method were used to identify the solid phases of the two ternary systems (KBr–$SrBr_2$–H_2O and NaBr–$SrBr_2$–H_2O) at 308 K.

2. Results and Discussion

2.1 Ternary KBr–$SrBr_2$–H_2O system at 308 K

The mass fraction of the components was determined by experimental analysis of the liquid phase and wet slag of the ternary KBr–$SrBr_2$–H_2O system at 308 K, as summarized in **Table 3**. By using the mass fraction data, a stable-phase diagram was constructed, as displayed in **Figure 1**, demonstrating that this ternary system is a simple co-saturated system. The diagram consists of two univariant curves, two crystalline regions, and one invariant point, and there is no double salt or solid solution. For the univariant curve A_1E_1, the line is relatively short: when the system is saturated with strontium bromide and a small amount of potassium bromide is added to the saturated solution of strontium bro-

Table 3 Solubility of salts in ternary KBr–$SrBr_2$–H_2O system at 308 K[a]

No.	Compositions of solution100·w(B)		Compositions of wet residue100·w(B)		Equilibrium solids
	w(KBr)	w($SrBr_2$)	w(KBr)	w($SrBr_2$)	
1, A_1	0.00	52.17	—	—	$SrBr_2·6H_2O$
2	1.81	50.88	0.60	64.85	$SrBr_2·6H_2O$
3	4.08	50.01	1.22	62.90	$SrBr_2·6H_2O$
4, E_1	6.99	47.85	12.75	54.82	KBr+$SrBr_2·6H_2O$
5	10.18	42.27	65.29	15.96	KBr
6	12.71	37.49	70.49	13.44	KBr
7	18.68	29.48	69.96	11.58	KBr
8	21.10	25.86	74.03	9.08	KBr
9	26.25	19.24	77.59	6.77	KBr
10	29.27	15.65	81.12	4.64	KBr
11	32.58	11.87	82.29	3.20	KBr
12	34.94	8.03	82.14	2.10	KBr
13	38.66	4.15	80.94	0.84	KBr
14	40.37	2.26	—	—	KBr
15, B_1	42.67	0	—	—	KBr

[a]Note: w(B) is the mass fraction of component B in the saturated solution. Standard uncertainty u: u(T) = 0.1 K, $u_r(w(KBr)) = 0.005$, $u_r(w(SrBr_2)) = 0.005$. Dash (—) indicates not detected.

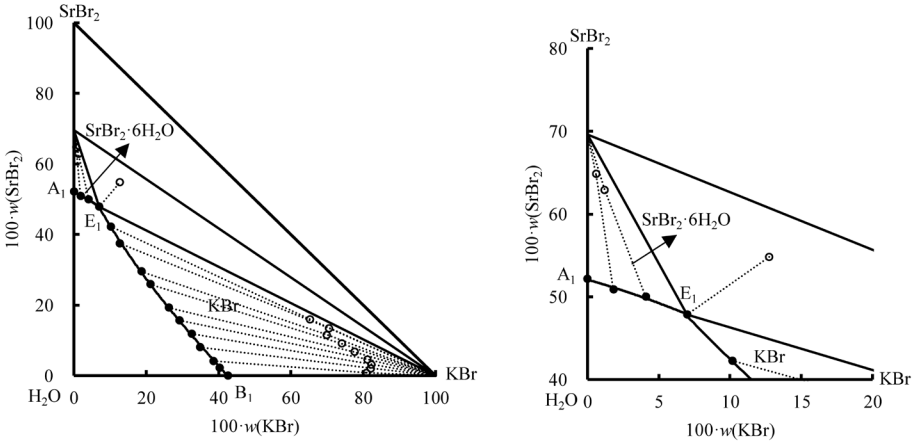

Fig. 1 Phase diagram and enlarged partial diagram for ternary KBr–$SrBr_2$–H_2O system at 308 K
○, Wet slag points; ●, liquid phase points

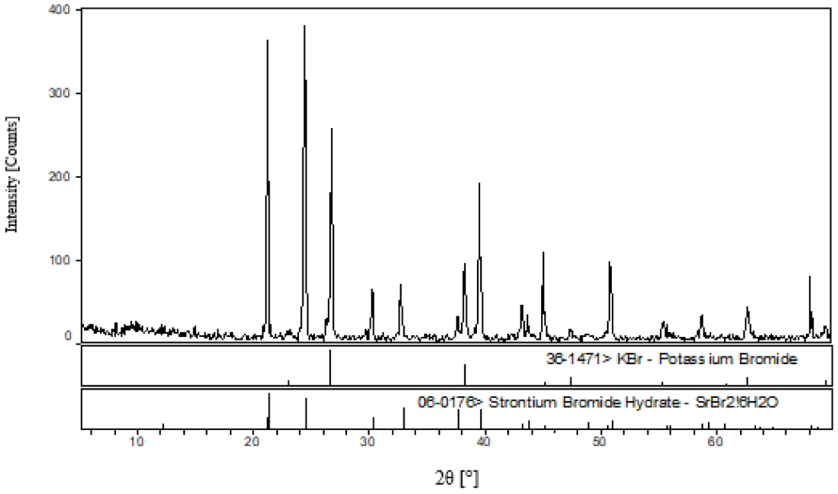

Fig. 2 X-ray diffraction pattern of invariant point E_1 ($KBr+SrBr_2\cdot 6H_2O$) in the ternary KBr–$SrBr_2$–H_2O system at 308 K

Table 4 Solubility of salts in ternary NaBr–$SrBr_2$–H_2O system at 308 K[a]

No.	Compositions of solution100·w(B)		Compositions of wet residue100·w(B)		Equilibrium solids
	w(NaBr)	$w(SrBr_2)$	w(NaBr)	$w(SrBr_2)$	
1, A_2	0.00	52.17	—	—	$SrBr_2\cdot 6H_2O$
2	2.08	50.23	0.96	62.02	$SrBr_2\cdot 6H_2O$
3	4.43	47.96	1.74	61.06	$SrBr_2\cdot 6H_2O$
4	5.82	47.13	1.98	61.02	$SrBr_2\cdot 6H_2O$
5	9.41	43.99	3.12	60.21	$SrBr_2\cdot 6H_2O$
6	14.10	40.35	4.01	60.74	$SrBr_2\cdot 6H_2O$
7	19.44	35.71	5.34	59.03	$SrBr_2\cdot 6H_2O$
8, E_2	22.45	33.32	25.27	41.81	$NaBr\cdot 2H_2O+SrBr_2\cdot 6H_2O$
9	23.80	31.26	55.78	11.71	$NaBr\cdot 2H_2O$
10	26.53	27.62	58.51	8.83	$NaBr\cdot 2H_2O$
11	29.88	23.76	60.46	7.12	$NaBr\cdot 2H_2O$
12	34.50	17.80	61.56	5.45	$NaBr\cdot 2H_2O$
13	38.46	13.64	63.39	4.26	$NaBr\cdot 2H_2O$
14	42.55	8.58	64.67	2.54	$NaBr\cdot 2H_2O$
15	46.63	4.57	67.31	1.13	$NaBr\cdot 2H_2O$
16	48.27	2.11	68.18	0.11	$NaBr\cdot 2H_2O$
17, B_2	50.73	0	—	—	$NaBr\cdot 2H_2O$

[a]Note: w(B) is the mass fraction of component B in the saturated solution. Standard uncertainty u: $u(T)=0.1$ K, $u_r(w(NaBr))=0.003$, $u_r(w(SrBr_2))=0.005$. Dash (—) indicates not detected.

mide, the system becomes saturated with the two salts. The univariant curve E_1B_1 is longer: when the system is saturated with potassium bromide and strontium bromide is added gradually, the mass fraction of potassium bromide gradually decreases and the mass fraction of strontium bromide increases, which means that strontium bromide has a strong salting-out effect on potassium bromide. Therefore, the solid phase crystallization zone of potassium bromide is much larger than that of strontium bromide, indicating that potassium bromide readily precipitates from solution. For the invariant point E_1 in Figure 1, the mass fraction of salt in the liquid phase is listed in Table 3. The solid phase at the invariant point E_1 was investigated by chemical analysis and it was found that potassium bromide and strontium bromide were both present at this point, the same was found from X-ray diffraction identification, as shown in **Figure 2**. From Figure 2, it is found that the peak position and intensity of the equilibrium solid phases at the invariant point E_1 are in good agreement with the standard card (X-ray powder diffraction pattern of peaks intensity and angle of pure single salt). Combined with the wet slag method, it was determined that the equilibrium solid phases are KBr and $SrBr_2\cdot 6H_2O$.

2.2 Ternary NaBr–$SrBr_2$–H_2O system at 308 K

Table 4 shows the compositions of the two salts in the liquid and wet solid phases of the ternary NaBr–$SrBr_2$–H_2O system at 308 K. Based on these solubility values, the equilibrium phase diagram was constructed, as shown in **Figure 3**. In the phase diagram, the univariant curves A_2E_2 and E_2B_2 are the solubility curves of the single salt $SrBr_2$ and

NaBr, respectively. The solid phase crystallization region of $NaBr·2H_2O$ is slightly larger than that of $SrBr_2·6H_2O$, further confirming that the solubility of NaBr is less than that of $SrBr_2$. In addition, there is only one invariant point in this system, and the corresponding solid phases are $NaBr·2H_2O$ and $SrBr_2·6H_2O$, and no compound salt or solid solution was found. The solid phase at invariant point E_2 was determined by X-ray diffraction, where the equilibrium solids $NaBr·2H_2O$ and $SrBr_2·6H_2O$ were identified, as presented in **Figure 4**. The position and intensity of the peak in Figure 4 are basically consistent with the standard card. It can be determined that the equilibrium solid phases are $NaBr·2H_2O$ and $SrBr_2·6H_2O$.

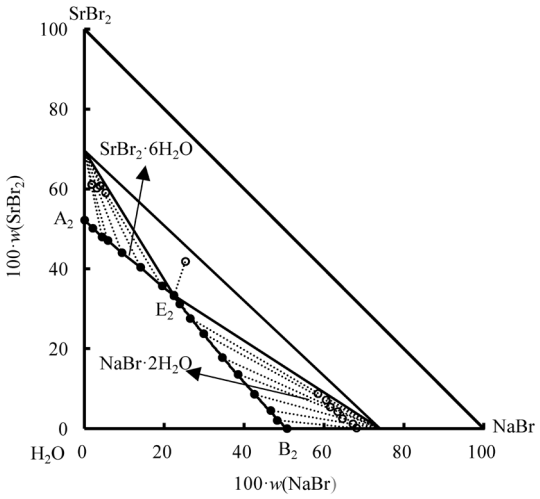

Fig. 3 Phase diagram of ternary $NaBr–SrBr_2–H_2O$ system at 308 K
◯, Wet slag points; ●, liquid phase points

3. Comparison and Discussion

3.1 Ternary $KBr–SrBr_2–H_2O$ system at different temperatures

Relevant research has been performed on this ternary system at different temperatures. For instance, Li *et al.* carried out a phase equilibrium and thermodynamic modeling study of the ternary $KBr–SrBr_2–H_2O$ system at 288 K. Our research group also measured the solubility of this ternary system at 323 and 348 K. In this work, the invariant points at different temperatures are summarized in **Table 5**, and comparative diagrams are plotted in **Figure 5**. The results demonstrate that the solubility trends of the ternary systems at different temperatures are the same, the mass fraction of potassium bromide is reduced incrementally, while the content of strontium bromide increases gradually. The solubility curves of strontium bromide are shorter than those of potassium bromide, and the crystallization zones of strontium bromide are smaller, while those of potassium bromide follow the opposite trend. These observations show that strontium bromide has a strong salting-out effect on potassium bromide, and the equilibrium solid phases corresponding to the invariant

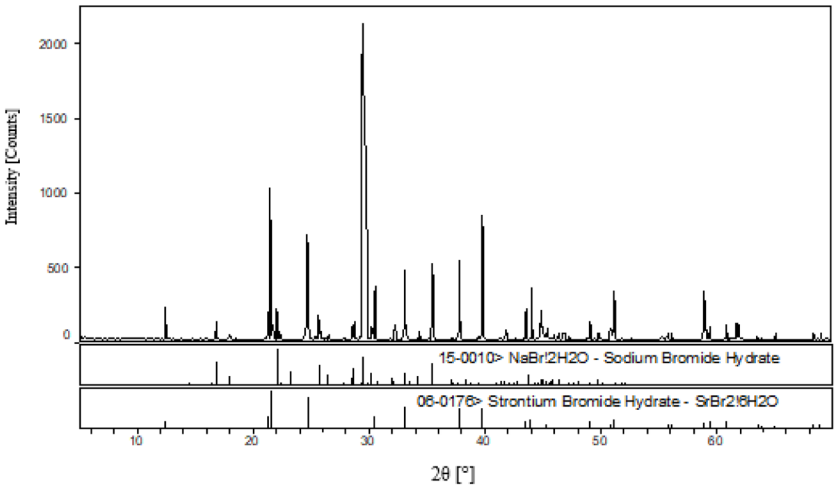

Fig. 4 X-ray diffraction pattern of invariant point E_2 ($NaBr+SrBr_2·6H_2O$) in the ternary $NaBr–SrBr_2–H_2O$ system at 308 K

Table 5 Solubility of salts at invariant points in ternary $KBr–SrBr_2–H_2O$ system at different temperatures

T/K	Compositions of liquid phase at the invariant points $100·w(B)^a$		Equilibrium solid phases [b]	Ref
	$w(KBr)$	$w(SrBr_2)$		
288	6.97	43.09	S6+K	Li *et al.* (2018)
308	6.99	47.85	S6+K	This work
323	7.03	50.76	S6+K	Cui *et al.* (2017)
348	6.41	55.19	S6+K	Cui (2017)

[a] $w(B)$: Mass fraction; [b] S6: $SrBr_2·6H_2O$, K: KBr

points all contain KBr and $SrBr_2 \cdot 6H_2O$ at these temperatures.

3.2 Ternary NaBr–$SrBr_2$–H_2O system at different temperatures

Similar to the ternary KBr–$SrBr_2$–H_2O system, the phase equilibria of the ternary NaBr–$SrBr_2$–H_2O system has also been evaluated at different temperatures. The specific data are presented in **Table 6**. As shown in **Figure 6**, at these temperatures, this ternary system is relatively simple with only one invariant point and two solubility curves. It is deduced from the univariant curves that as the mass fraction of sodium bromide increased, the mass fraction of strontium bromide decreased, and the same solid phase ($SrBr_2 \cdot 6H_2O$) was present in all crystallization zones. However, at 348 K, the solid phase changed from $NaBr \cdot 2H_2O$ to NaBr, which indicates that at higher temperatures, the solid phase can be dehydrated to anhydrous sodium bromide, and the crystallization area of NaBr is much larger than that of $SrBr_2 \cdot 6H_2O$. The results confirm that strontium bromide has a strong salting-out effect on sodium bromide.

Conclusions

Ternary NaBr–$SrBr_2$–H_2O and KBr–$SrBr_2$–H_2O systems were studied herein via a three-pronged approach: (1) Research methods based on isothermal dissolution equilibrium and chemical analysis were employed. (2) Experimental data for the two relatively simple ternary systems at 308 K were obtained via experiment, demonstrating that no double salts or solid solutions were formed, and both include one invariant point, two solubility curves, and two solid-phase crystallization regions, all of which contain solid-phase $SrBr_2 \cdot 6H_2O$. The crystalline zones of solid-phase $SrBr_2 \cdot 6H_2O$ in the two systems are small, indicating that strontium bromide has a salting-out effect on potassium bromide and sodium bromide, respectively. (3) The phase diagrams of each of the two ternary systems at different temperatures were compared, demonstrating that the trends in the phase diagrams of the two systems are very similar, except that the solid-phase sodium bromide in the ternary NaBr–$SrBr_2$–H_2O system at 348 K underwent dehydration from $NaBr \cdot 2H_2O$ to NaBr.

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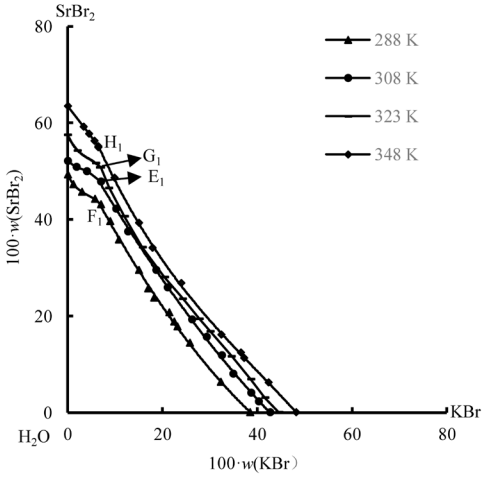

Fig. 5 Equilibrium phase diagrams of ternary KBr–$SrBr_2$–H_2O system at different temperatures

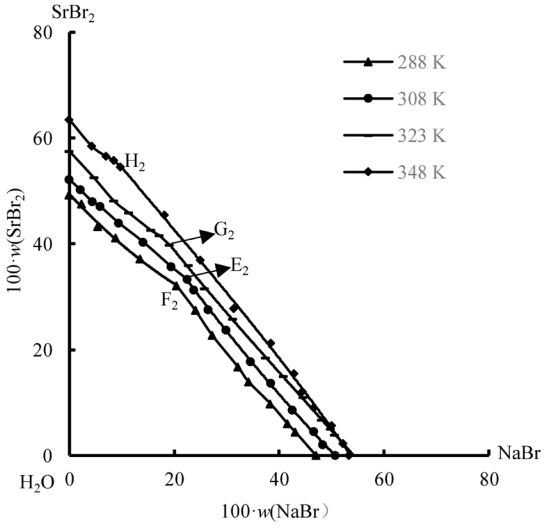

Fig. 6 Equilibrium phase diagrams of ternary NaBr–$SrBr_2$–H_2O system at different temperatures

Table 6 Solubility of salts at invariant points in the ternary NaBr–$SrBr_2$–H_2O system at different temperatures

T [K]	Compositions of liquid phase at the invariant points $100 \cdot w(B)$ [a]		Equilibrium solid phases [b]	Ref
	w(NaBr)	w($SrBr_2$)		
288	20.49	32.08	S6+N2	Li *et al.* (2018)
308	22.45	33.32	S6+N2	This work
323	19.12	39.78	S6+N2	Cui *et al.* (2017)
348	9.76	54.53	S6+N	Cui (2017)

[a] w(B): Mass fraction; [b] S6: $SrBr_2 \cdot 6H_2O$, N: NaBr, N2: $NaBr \cdot 2H_2O$.

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