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Measurements of (Solid + Liquid) Phase Equilibria in the Quaternary System NaBr + KBr + $SrBr_2$ + H_2O and Two Subsystems NaBr + $SrBr_2$ + H_2O and KBr + $SrBr_2$ + H_2O at T = 323 K

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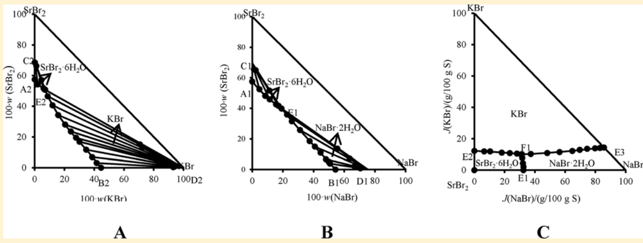
ABSTRACT: The (solid + liquid) phase equilibria of the NaBr + KBr + $SrBr_2$ + H_2O quaternary system and two subsystems (NaBr + $SrBr_2$ + H_2O and KBr + $SrBr_2$ + H_2O) were studied at T = 323 K using the isothermal dissolution method. With the solubility values of each system, the stable phase diagrams of these systems were drawn. It shows that there is neither solid solution nor double salt in the diagrams. The three systems are all of the hydrate type I, and only one invariant point is found in the phase diagrams of the three systems.



1. INTRODUCTION

In China, there are many salt lakes and underground brines. With the deep development of exploration and scientific research, more and more underground brine resources were discovered and developed. The underground brine resources in China's Sichuan Basin are the most important liquid mineral resources and the most famous in the world because of the big basin area, huge brine resource reserve, rich resources, and varieties of useful components such as boron, potassium, bromine, strontium iodine, and lithium, and all of which exceed the level of industry exploitation and utilization. Especially, the potassium, bromine, and strontium contents are up to 53.3, 2.533, and 0.166 $g \cdot L^{-1}$, respectively.[1,2]

The underground brine containing strontium of Sichuan Basin mostly comes from 50−3000 m deep underground. The pressure is about 45 MPa in formation depth of 3000 m. The average temperature of Sichuan Basin is 15 °C, the temperature of superficial brine is generally about 298 K. The temperature of most deep brine is about 323−348 K. For example, the temperature of underground brine about 3260 m deep in Xuanda Basin is 362 K.[3] The exploitation of underground brine mineral resources can be accelerated with the help of the exploitation of oil-gas fields in Sichuan Basin. Brine can flow out of the drill automatically with the aid of pressure, so the exploitation is very convenient.

The phase equilibria study results of brine systems can predict the regularity of crystallizing-out of saline minerals, which is the foundation of brine comprehensive application. Some development of salt lakes and brines such as the Qarhan Salt Lake in China, Sears salt lake and Great Salt Lake in America, the Dead Sea in Israel, and Artakama in Chile are based on the related phase equilibria and phase diagram study results of brine systems.[4]

So far, the Sr-bearing system of underground brine in Sichuan Basin has not been researched systematically. Regarding the Sr-bearing phase equilibria, a series of efforts have been made that have mainly concentrated on chloride systems: quinary systems LiCl + NaCl + KCl + $SrCl_2$ + H_2O and NaCl + KCl + $SrCl_2$ + $CaCl_2$ + H_2O and the subsystems.[5−14] At present, for the underground brine in Sichuan Basin, our group is carrying out research in the $SrBr_2$-bearing system mainly. The phase equilibria of quaternary systems NaBr + $SrBr_2$ + $MgBr_2$ + H_2O and KBr + $SrBr_2$ + $MgBr_2$ + H_2O at T = 323 K and NaBr + KBr + $SrBr_2$ + H_2O at T = 348 K have been published.[15,16] The NaBr + KBr + $SrBr_2$ + H_2O quaternary system is a subsystem of the underground brines. A study on the phase equilibria of this system and relevant Sr-bearing subsystems at T = 323 K has not been reported, which is the object of this paper.

2. EXPERIMENTAL SECTION

2.1. Reagents and Apparatus. The chemicals used are analytic reagents and are listed in Table 1. They included sodium bromide, potassium bromide, magnesium bromide hexahydrate, and strontium bromide hexahydrate. The water used was deionized water (its pH was about 6.60, and its electrical conductivity was less than 1.20 × 10^{-4} $S \cdot m^{-1}$ at indoor temperature).

A thermostated oscillator (HZS-H type) was made by Harbin DongLian Electronic Technology Co., Ltd. and used in the solid−liquid equilibria experiments. After secondary calibration, its precision of temperature control is ±0.1 K. A

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Table 1. Chemicals Used

chemical name	source	initial mass fraction purity	purification method	analysis method
KBr	Chengdu KeLong Chemical Reagent Factory	0.99	none	titration
NaBr	Chengdu KeLong Chemical Reagent Factory	0.99	none	titration
$SrBr_2 \cdot 6H_2O$	Shanghai Haorui Chemical Limited Corporation	0.99	none	titration

DX-2700 X-ray powder crystal diffraction analyzer supplied by Dandong Fangyuan Instrument Co., Ltd. was employed to identify the equilibrium solid phases.

2.2. Experimental Method. Experiments were done using the isothermal solution method. According to a certain proportion, a kind of new salt was put generally on the equilibrium composition of each invariant point for subsystems. Each univariant curve was given 4−10 groups, and then an appropriate amount of distilled water was added into each group of mixtures, respectively. The matched solution for experiments were put into ground glass bottles (200 mL, 4 cm in diameter, 16 cm high), sealed with plastic wrap, and then placed in a constant temperature bath oscillator (323 ± 0.1) K, oscillating continuously until balanced. Experimental equilibrium takes about 20 days; at the end of the reaction time, the supernatant liquid was removed from the flask every day, and chemical analysis was conducted. If there was no change in chemical composition for three consecutive days, the reaction was regarded in being in the equilibrium state. After equilibrium, the supernatant was used to determine the chemical composition of equilibrium liquid phase with the chemical analysis method.

2.3. Analytical Methods. The potassium ion concentration was measured using sodium tetraphenyl borate−quaternary ammonium salt back-titration with an indicator of titan yellow within ±0.5%. The strontium ion concentration was measured at pH 10 using titration with EDTA standard solution with an indicator of eriochrome black T within ±0.3%. The bromide ion concentration was measured using silver nitrate volumetric analysis with an indicator of potassium chromate within ±0.3%, and the sodium ion concentration was obtained using ion balance subtraction.

3. RESULTS AND DISCUSSION

3.1. Ternary System ($NaBr + SrBr_2 + H_2O$). Table 2 lists the measured values of solubility for $NaBr + SrBr_2 + H_2O$ system at T = 323 K. The solubility of equilibrated liquid phase is expressed in weight percent w(B). Figure 1 is the equilibrium phase diagram of the system which is plotted according to the data in Table 2. Figure 2 is the X-ray powder diffraction pattern of the invariant point E1 in this system. According to this figure, point E1 corresponds to the saturation point of $NaBr \cdot 2H_2O$ and $SrBr_2 \cdot 6H_2O$.

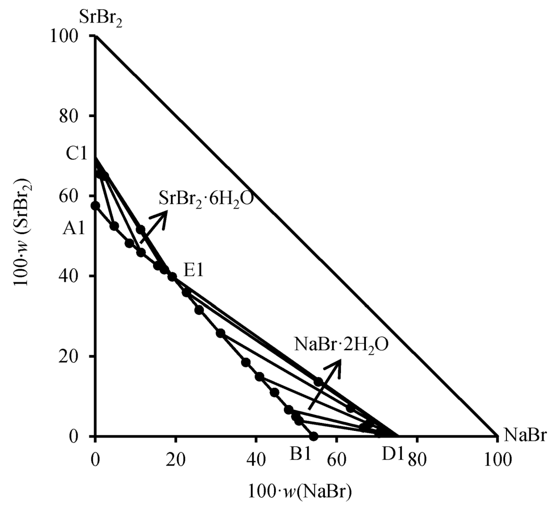

Figure 1. Phase diagram of the ternary system $NaBr + SrBr_2 + H_2O$ at T = 323 K and p = 0.1 MPa.

Table 2. Solubilities of Solution in the Ternary System $NaBr + SrBr_2 + H_2O$ at T = 323 K and p = 0.1 MPa[a]

no.	composition of liquid phase $100 \cdot w$(B) w(NaBr)	composition of liquid phase $100 \cdot w$(B) $w(SrBr_2)$	composition of wet residue $100 \cdot w$(B) w(NaBr)	composition of wet residue $100 \cdot w$(B) $w(SrBr_2)$	equilibrium phase solids
1, B1	54.27	0.00			$NaBr \cdot 2H_2O$
2	50.66	3.87	70.47	0.77	$NaBr \cdot 2H_2O$
3	49.86	4.94			$NaBr \cdot 2H_2O$
4	48.06	6.64	66.65	2.13	$NaBr \cdot 2H_2O$
5	44.53	10.96			$NaBr \cdot 2H_2O$
6	40.83	14.89	68.15	3.22	$NaBr \cdot 2H_2O$
7	37.41	18.47			$NaBr \cdot 2H_2O$
8	31.12	25.78	63.48	7.07	$NaBr \cdot 2H_2O$
9	25.80	31.51			$NaBr \cdot 2H_2O$
10	22.69	35.87	55.44	13.58	$NaBr \cdot 2H_2O$
11, E1	19.12	39.78			$NaBr \cdot 2H_2O + SrBr_2 \cdot 6H_2O$
12	17.15	41.57	11.24	51.55	$SrBr_2 \cdot 6H_2O$
13	15.54	42.59			$SrBr_2 \cdot 6H_2O$
14	11.38	45.84	2.23	64.92	$SrBr_2 \cdot 6H_2O$
15	8.49	48.13			$SrBr_2 \cdot 6H_2O$
16	4.70	52.47	1.16	65.44	$SrBr_2 \cdot 6H_2O$
17, A1	0.00	57.53			$SrBr_2 \cdot 6H_2O$

[a]Standard uncertainties u are $u(T)$ = 0.1 K, $u(p)$ = 0.005 MPa, $u[w(Na^+)]$ = 0.005, $u[w(Sr^{2+})]$ = 0.003, and $u[w(Br^-)]$ = 0.003.

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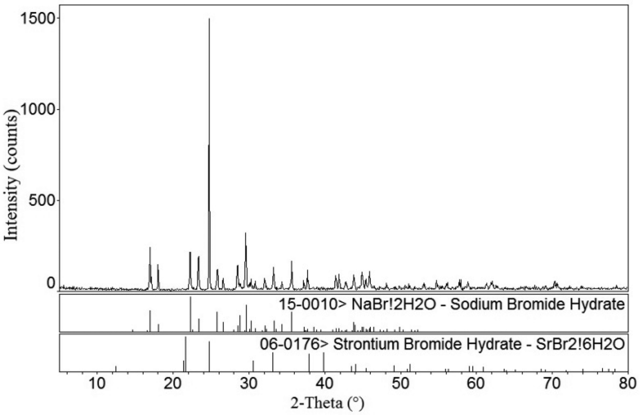

Figure 2. X-ray diffraction photograph of the invariant point E1 of the ternary system NaBr + $SrBr_2$ + H_2O at 323 K (NaBr·$2H_2O$ + $SrBr_2$·$6H_2O$).

In Figure 1, there are two crystallization regions of equilibrium solids: sodium bromide dihydrate (NaBr·$2H_2O$) and strontium bromide hexahydrate ($SrBr_2$·$6H_2O$). The crystallization region of $SrBr_2$·$6H_2O$ (A1E1C1) is small, whereas that of the salt NaBr·$2H_2O$ (B1E1D1) is large. At T = 323 K, this system (NaBr + $SrBr_2$ + H_2O) has one eutectic point, denoted as E1. Point A1 is the single salt saturation point of $SrBr_2$, and point B1 is that of NaBr with mass percentages of 57.53 and 54.27, respectively. The solubility of sodium bromide at 323 K has been reported in multiple references,[17−19] and the value is generally between 53.70 and 54.10. The deviation could be mainly caused by the temperature change, the purity of chemicals, the judgment of titration end point, or the volume of flask volume, pipet, and buret.

This system has two univariant curves represented by curves A1E1 and B1E1. At eutectic point E1, the system is saturated with two salts, $SrBr_2$·$6H_2O$ and NaBr·$2H_2O$, and its equilibrium liquid phase compositions are $w(SrBr_2) = 0.3978$ and $w(NaBr) = 0.1912$, respectively. Ternary system NaBr + $SrBr_2$ + H_2O is also a subsystem of quaternary system NaBr + $MgBr_2$ + $SrBr_2$ + H_2O, which has been reported at 323 K by our group.[15] In the literature, ternary system NaBr + $SrBr_2$ + H_2O has only one invariant point at 323 K, which is saturated with $SrBr_2$·$6H_2O$ and NaBr·$2H_2O$ [$w(SrBr_2) = 0.3946$, $w(NaBr) = 0.1920$]. The measured data in this paper are basically in agreement with the data reported by the references.

3.2. Ternary System (KBr + $SrBr_2$ + H_2O). Experimentally measured values of the solubility of the KBr + $SrBr_2$ + H_2O system at T = 323 K are presented in Table 3. Corresponding phase diagram at T = 323 K was plotted using these solubility data, which is shown in Figure 3.

Figure 3 shows that it contains two crystallization fields, two univariant curves, and one eutectic point. Neither solid solution nor double salt was found in KBr + $SrBr_2$ + H_2O system at T = 323 K. The X-ray powder diffraction pattern of the invariant point (point E2) is given in Figure 4. According to Figure 4, the two crystallization fields are anhydrous salt KBr and hydrous salt $SrBr_2$·$6H_2O$, respectively. The two univariant curves of this system are represented by curves A2E2 and B2E2. The crystallization region of hydrate $SrBr_2$·$6H_2O$ (A2E2C2) is small, whereas that of single salt KBr (B2E2D2) is large. Point A2 represents the saturation point of the single salt $SrBr_2$, and point B2 represents that of KBr. The experimental solubility data of KBr at 323 K are mass percentages of 44.60, which is consistent with reference experimental data (44.48−44.90) in the range of experimental errors.[17,20,21] Point E2 is the eutectic point that corresponds to the saturation of two salts: KBr and $SrBr_2$·$6H_2O$. Its equilibrium liquid phase composition is as follows: $w(KBr) = 0.0703$ and $w(SrBr_2) = 0.5076$, which is consistent with the literature values.[15]

Table 3. Solubilities of Solution in the Ternary System KBr + $SrBr_2$ + H_2O at T = 323 K and p = 0.1 MPa[a]

	composition of liquid phase 100·w(B)		composition of wet residue 100·w(B)		
no.	w(KBr)	w($SrBr_2$)	w(KBr)	w($SrBr_2$)	equilibrium phase solids
1, B2	44.60	0.00			KBr
2	41.65	3.06	93.12	0.38	KBr
3	38.67	6.86	93.95	0.68	KBr
4	34.59	11.67	94.30	1.09	KBr
5	30.12	16.82	93.50	1.53	KBr
6	27.83	19.43	85.96	3.89	KBr
7	24.30	23.57	90.39	2.99	KBr
8	20.46	28.05	87.50	4.45	KBr
9	15.77	34.24	82.64	7.15	KBr
10	12.00	40.63	72.36	13.03	KBr
11	8.76	46.54	70.68	15.13	KBr
12, E2	7.03	50.76			KBr + $SrBr_2$·$6H_2O$
13	6.80	50.79	4.53	57.15	$SrBr_2$·$6H_2O$
14	5.96	51.68	1.12	66.20	$SrBr_2$·$6H_2O$
15	2.13	54.29	0.13	68.51	$SrBr_2$·$6H_2O$
16, A2	0.00	57.53			$SrBr_2$·$6H_2O$

[a]Standard uncertainties u are $u(T) = 0.1$ K, $u(p) = 0.005$ MPa, $u[w(K^+)] = 0.005$, $u[w(Sr^{2+})] = 0.003$, $u[w(Br^-)] = 0.003$.

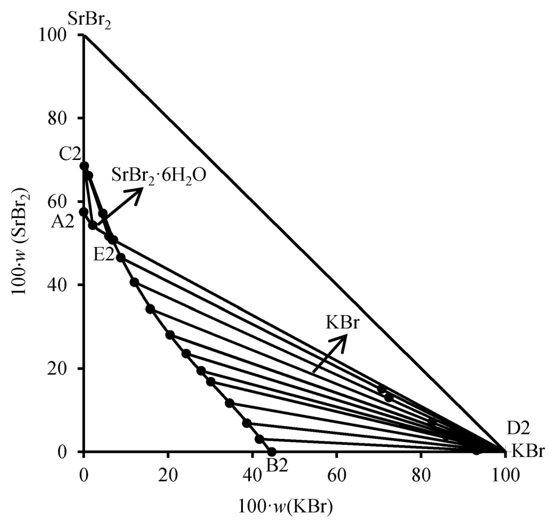

Figure 3. Phase diagram of the ternary system KBr + $SrBr_2$ + H_2O at T = 323 K and p = 0.1 MPa.

3.3. Quaternary System (NaBr + KBr + $SrBr_2$ + H_2O). Quaternary system NaBr + KBr + $SrBr_2$ + H_2O consists of three ternary subsystems: (1) NaBr + KBr + H_2O, (2) NaBr + $SrBr_2$ + H_2O, and (3) KBr + $SrBr_2$ + H_2O. The three Br-bearing ternary subsystems at T = 323 K are all hydrate type I. The isothermal phase diagram of each system consists of two solid phase regions, two univariant curves, and one invariant point.

The experimental solubility data of NaBr + KBr + $SrBr_2$ + H_2O system at T = 323 K are presented in Table 4, and the

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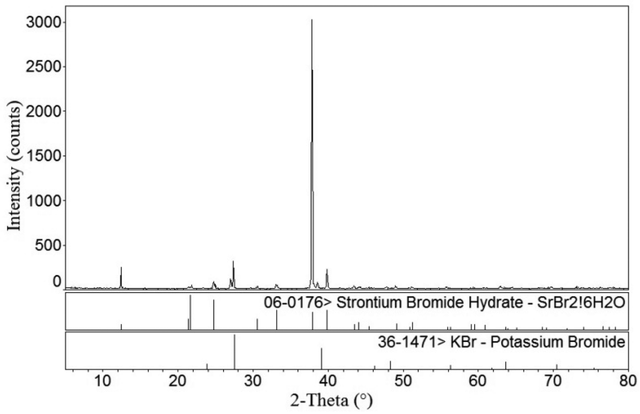

Figure 4. X-ray diffraction photograph of the invariant point E2 of the ternary system KBr + $SrBr_2$ + H_2O at 323 K (KBr + $SrBr_2{\cdot}6H_2O$).

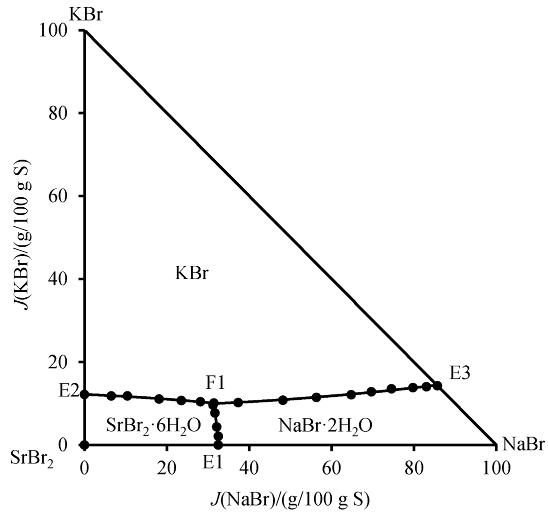

Figure 5. Phase diagram of the quaternary system NaBr + KBr + $SrBr_2$ + H_2O at T = 323 K and p = 0.1 MPa.

composition of the equilibrium liquid phase is expressed as mass percent. The phase diagram of NaBr + KBr + $SrBr_2$ + H_2O system is drawn with Jänecke indices ($J(NaBr) + J(KBr) + J(SrBr_2) = 100$ g) by the data in Table 4, as shown in Figure 5 with solid lines.

The phase diagram of NaBr + KBr + $SrBr_2$ + H_2O system at T = 323 K has three crystallization fields of single salts, one invariant point, and three univariant curves. The three solid crystallization fields are $SrBr_2{\cdot}6H_2O$, $NaBr{\cdot}2H_2O$, and KBr, respectively. The solid crystalline phase region of anhydrous salt KBr is the largest, and hydrous salt $SrBr_2{\cdot}6H_2O$ is smallest. It shows that the solubility of KBr is lowest, so it is the easiest to crystallize in the quaternary system. Except for the three original compositions, neither double salt nor solid solution was found in the quaternary system. The number of crystal water of each hydrous salt is also unchanged. The three solubility curves of the system are E1F1, E2F1, and E3F1. The only one invariant point is labeled as F1.

The X-ray powder diffraction photographs of the point F1 are given in Figure 6, where point F1 is the saturation point of $SrBr_2{\cdot}6H_2O$, KBr, and $NaBr{\cdot}{}_2H_2O$. The liquid phase composition of invariant point F1 is $w(SrBr_2)$ = 0.3527, w(KBr) = 0.0608, and w(NaBr) = 0.1896.

Figure 7 is the water content figure of NaBr + KBr + $SrBr_2$ + H_2O system at T = 323 K. The water content first decreases at the univariant curve E2F1 and then increases at the univariant curve F1E5 with an increase of the Jänecke index values of J(NaBr).

Table 4. Solubilities of Solution in the Quaternary System NaBr + KBr + $SrBr_2$ + H_2O at T = 323 K and p = 0.1 MPa[a]

	composition of solution $w(B)\times100$			Jänecke index J/(g/100 g), $J(NaBr)+J(KBr)+J(SrBr_2)$ = 100 g				
no.	w(NaBr)	w(KBr)	$w(SrBr_2)$	J(NaBr)	J(KBr)	$J(SrBr_2)$	$J(H_2O)$	equilibrium solids
1, E3	48.58	8.10	0.00	85.70	14.30	0.00	76.43	NB2 + KB
2	47.32	8.01	1.67	83.02	14.06	2.92	75.44	NB2 + KB
3	45.94	7.96	3.70	79.75	13.82	6.43	73.60	NB2 + KB
4	42.47	7.70	6.79	74.55	13.52	11.93	75.55	NB2 + KB
5	40.28	7.42	10.15	69.64	12.82	17.54	72.87	NB2 + KB
6	36.82	6.88	13.15	64.77	12.10	23.13	75.88	NB2 + KB
7	32.67	6.64	18.71	56.30	11.45	32.25	72.35	NB2 + KB
8	28.41	6.36	24.17	48.20	10.80	41.00	69.66	NB2 + KB
9	22.50	6.15	31.63	37.32	10.20	52.48	65.91	NB2 + KB
10, F1	18.96	6.08	35.27	31.44	10.09	58.47	65.78	NB2 + KB + SB
11, E1	19.12	0.00	39.78	32.46	0.00	67.54	69.78	NB2 + SB
12	19.04	1.23	38.28	32.52	2.10	65.38	70.81	NB2 + SB
13	18.93	2.57	37.49	32.09	4.36	63.55	69.51	NB2 + SB
14	18.79	4.58	35.93	31.68	7.72	60.59	68.62	NB2 + SB
15	18.74	5.78	35.55	31.19	9.63	59.18	66.48	NB2 + SB
16, E2	0.00	7.03	50.76	0.00	12.16	87.84	73.04	KB + SB
17	3.79	6.87	47.43	6.52	11.83	81.65	72.14	KB + SB
18	6.13	6.94	45.63	10.44	11.83	77.73	70.37	KB + SB
19	10.69	6.53	41.77	18.12	11.08	70.81	69.52	KB + SB
20	14.05	6.41	39.32	23.50	10.72	65.78	67.27	KB + SB
21	16.89	6.26	36.85	28.16	10.43	61.41	66.68	KB + SB

[a]Abbreviations: NB2 = $NaBr{\cdot}2H_2O$, KB = KBr, SB = $SrBr_2{\cdot}6H_2O$. Standard uncertainties u are $u(T)$ = 0.1 K, $u(p)$ = 0.005 MPa, $u[w(Na^+)]$ = 0.005, $u[w(K^+)]$ = 0.005, $u[w(Sr^{2+})]$ = 0.003, and $u[w(Br^-)]$ = 0.003.

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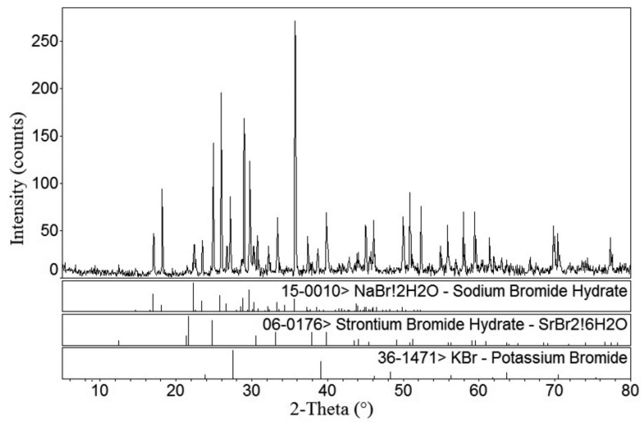

Figure 6. X-ray diffraction photograph of the invariant point F1 of the quaternary system NaBr + KBr + $SrBr_2$ + H_2O at 323 K ($NaBr\cdot 2H_2O$ + $SrBr_2\cdot 6H_2O$ + KBr).

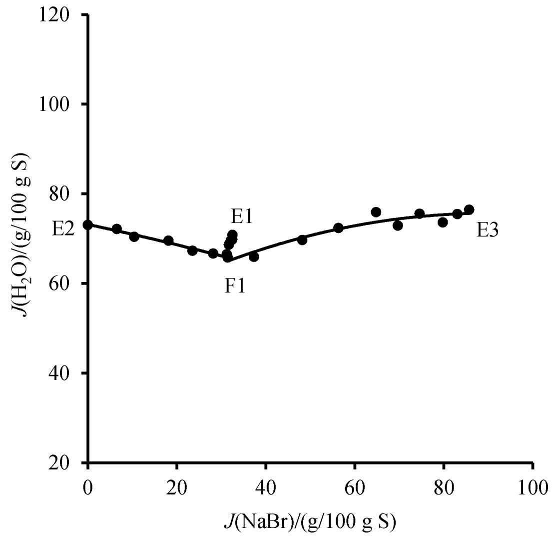

Figure 7. Water contents of saturated solutions in quaternary system NaBr + KBr + $SrBr_2$ + H_2O at T = 323 K and p = 0.1 MPa.

4. CONCLUSIONS

Measurements of solid + liquid equilibria of system NaBr + KBr + $SrBr_2$ + H_2O and the subsystems NaBr + $SrBr_2$ + H_2O and KBr + $SrBr_2$ + H_2O were investigated at T = 323 K using an isothermal solubility method. In these three systems, there is neither double salt nor solid solution.

At T = 323 K, the phase diagrams of NaBr + $SrBr_2$ + H_2O and KBr + $SrBr_2$ + H_2O systems all have two solid phase crystallization areas, two solubility curves, and only one invariant point. In the NaBr + $SrBr_2$ + H_2O system, the fields of crystallization are $NaBr\cdot 2H_2O$ and $SrBr_2\cdot 6H_2O$, and those in the KBr + $SrBr_2$ + H_2O system are KBr and $SrBr_2\cdot 6H_2O$.

In the phase diagram of the NaBr + KBr + $SrBr_2$ + H_2O quaternary system, only one invariant point F1, three univariate curves E1F1, E2F1, E3F1, and three crystalline zones $SrBr_2\cdot 6H_2O$, $NaBr\cdot 2H_2O$, and KBr were found at T = 323 K.

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Notes

The authors declare no competing financial interest.

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