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Solid−Liquid Equilibria in the Quaternary Systems NaBr−SrBr$_2$−MgBr$_2$−H$_2$O and KBr−SrBr$_2$−MgBr$_2$−H$_2$O at 323 K

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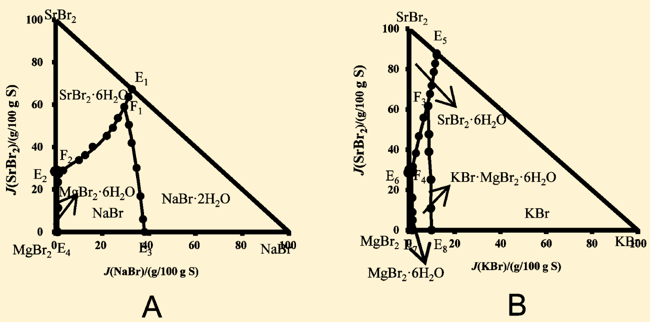
ABSTRACT: The two quaternary systems NaBr−SrBr$_2$−MgBr$_2$−H$_2$O and KBr−SrBr$_2$−MgBr$_2$−H$_2$O at 323 K were constructed by the isothermal dissolution equilibrium method. The mass fraction of every component of the equilibrium liquid phase in the two systems at 323 K was calculated. The phase diagrams and water content diagrams were plotted in accordance with the Jañecke indices of salts and water. The system NaBr−SrBr$_2$−MgBr$_2$−H$_2$O at 323 K has two invariant points, five univariant curves, and four solid phase crystallization areas, which correspond to strontium bromide hexahydrate (SrBr$_2$·6H$_2$O), magnesium bromide hexahydrate (MgBr$_2$·6H$_2$O), sodium bromide (NaBr), and sodium bromide dihydrate (NaBr·2H$_2$O). The system KBr−SrBr$_2$−MgBr$_2$−H$_2$O at 323 K has double salt (KBr·MgBr$_2$·6H$_2$O) without solid solution formation. It has two invariant points, five univariant curves, and four solid phase crystallization areas corresponding to strontium bromide hexahydrate (SrBr$_2$·6H$_2$O), magnesium bromide hexahydrate (MgBr$_2$·6H$_2$O), double salt (KBr·MgBr$_2$·6H$_2$O), and potassium bromide (KBr).



1. INTRODUCTION

In recent years, it was found that the quality of underground brines of Sichuan basin is stable. The underground brines have many useful components, for example, K$^+$, B^{3+}, Na$^+$, Li$^+$, Sr^{2+}, and Mg^{2+}, and so forth.[1] The underground brines of Sichuan basin have recently attracted much attention for their use in a wide range of applications, especially in the fields of metallurgy, chemical industry, and medicine. If the resources can be developed and used effectively, it can greatly relieve the present situation of resources shortage in our country.[2] It was necessary for us to analyze the equilibrium relationship of the useful components in underground brine.

So far, a range of research about the water−salt system have been finished, which the strontium is involved in, such as the Na−K−Sr−Cl−H$_2$O system and its subsystems Na−Sr−Cl−H$_2$O and K−Sr−Cl−H$_2$O at 291, 323, and 373 K[3] and the systems Na−K−Sr−Ca−Cl−H$_2$O at 291, 309.5, and 373 K.[4] The phase equilibria of ternary systems Ca−Sr−Cl−H$_2$O between 291 and 386 K,[5] Na−Sr−Cl−H$_2$O and K−Sr−Cl−H$_2$O at 298 K, Mg−Sr−Cl−H$_2$O at 292, 353, and 373 K have also been completed.[6−8] Furthermore, the ternary systems Na−Mg−Br−H$_2$O and K−Mg−Br−H$_2$O at 323 K also have been done.[9,10]

In addition, a large amount of the related phase equilibrium systems have been performed by our group, for instance, the ternary systems Mg−Sr−Cl−H$_2$O at 323 and 348 K,[11] the systems Na−Sr−Cl−H$_2$O and K−Sr−Cl−H$_2$O at 348 K,[12] and K−Sr−Cl−H$_2$O and Na−K−Sr−Cl−H$_2$O at 323 K.[13] The systems K−Ca−Br−H$_2$O and Na−Ca−Br−H$_2$O at 348.15 K,[14] Na−K−Ca−Br−H$_2$O at 298 K,[15] and Na−K−Br−B$_4$O$_7$−H$_2$O at 298 K[16] have been done.

The solid−liquid equilibrium of quaternary systems NaBr−SrBr$_2$−MgBr$_2$−H$_2$O and KBr−SrBr$_2$−MgBr$_2$−H$_2$O at 323 K have not been reported up to now. The research on the two systems is an important supplement for the multiple phase equilibria, which also can instruct us to extract the strontium salt and potassium salt.

2. EXPERIMENTAL SECTION

2.1. Reagents and Instruments. The experimental reagents which were involved in the experiment are listed in Table 1. The distilled water used in the experiment was produced by the water polishing system. When the conductivity is lower than 1×10^{-5} S·m^{-1} and pH = 6, the water reaches the usage standard.

An HZS−HA type thermostatic water bath oscillator with the precision of ±0.1 K was employed in the experiment.

A standard analytical balance (AL104) of 110 g capacity produced by the Mettler Toledo Instruments Co., Ltd. was employed in weighing the mass of the solution.

2.2. Experimental Method. Experiments here were constructed by the isothermal solution equilibrium method. The third salt was added to the ternary subsystem at the invariant points of the pre-existing salts at 323 K. The plastic

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Table 1. Analytical Experimental Reagents

chemical name	initial purity (w/w %)	purification method	source
magnesium bromide hexahydrate ($MgBr_2·6H_2O$)	98.0	chemical analysis	Shanghai Xinbao Fine Chemical Plant
strontium bromide hexahydrate ($SrBr_2·6H_2O$)	99.0	chemical analysis	Shanghai Haorui Chemical Limited Corporation, China
sodium bromide (NaBr)	99.0	chemical analysis	Chengdu Kelong Chemical Reagent Factory
potassium bromide (KBr)	99.0	chemical analysis	Chengdu Kelong Chemical Reagent Factory

Table 2. Experimental Values of Solubilities and Jänecke Indices of Dry Salt (J) of the Quaternary System $NaBr-SrBr_2-MgBr_2-H_2O$ at 323 K and Pressure p = 0.1 MPa[a,b]

	composition of solution w(B) × 100			Jänecke index J (g/100 g) J(NaBr) + J($MgBr_2$) + J($SrBr_2$) = 100 g				
no.	w(NaBr)	w($MgBr_2$)	w($SrBr_2$)	J(NaBr)	J($MgBr_2$)	J($SrBr_2$)	J(H_2O)	equilibrium solids
1, E_1	19.20	0.00	39.46	32.73	0.00	67.27	70.49	NB2 + SB
2	18.24	3.06	37.09	31.24	5.24	63.52	71.28	NB2 + SB
3, F_1	16.96	6.70	33.95	29.44	11.62	58.94	73.59	NB2 + SB + NB
4	15.50	11.39	31.13	26.71	19.63	53.66	72.35	NB + SB
5	14.22	15.24	28.31	24.61	26.38	49.01	73.09	NB + SB
6	12.57	18.79	25.88	21.95	32.82	45.22	74.71	NB + SB
7	9.07	24.94	22.82	15.96	43.88	40.15	75.93	NB + SB
8	7.07	28.55	20.16	12.68	51.18	36.14	79.25	NB + SB
9	5.66	31.52	19.03	10.07	56.08	33.85	77.94	NB + SB
10	1.85	37.82	16.18	3.32	67.71	28.97	79.04	NB + SB
11, E_2	0.00	39.81	15.90	0.00	71.46	28.54	79.51	MB + SB
12	0.22	39.39	15.88	0.39	70.99	28.62	80.22	MB + SB
13	0.38	39.12	15.87	0.68	70.66	28.66	80.61	MB + SB
14, F_2	0.53	39.41	15.35	0.95	71.28	27.77	80.87	MB + SB + NB
15, E_4	0.51	51.72	0.00	0.98	99.02	0.00	91.45	NB + MB
16	0.54	47.64	6.15	0.99	87.69	11.32	84.05	NB + MB
17	0.54	41.82	13.00	0.98	75.54	23.48	80.64	NB + MB
18, E_3	17.37	28.22	0.00	38.10	61.90	0.00	119.33	NB + NB2
19	18.02	27.20	2.89	37.45	56.54	6.01	107.87	NB + NB2
20	17.49	22.49	8.08	36.39	46.80	16.81	108.05	NB + NB2
21	17.91	18.12	15.56	34.71	35.12	30.17	93.82	NB + NB2
22	17.34	13.54	22.29	32.62	25.46	41.93	88.06	NB + NB2
23	17.98	10.35	28.93	31.40	18.07	50.53	74.64	NB + NB2

[a]NB2, $NaBr·2H_2O$; NB, NaBr; MB, $MgBr_2·6H_2O$; SB, $SrBr_2·6H_2O$. [b]Standard uncertainties u are $u(T)$ = 0.1 K, $u_r(p)$ = 0.05, $u(w(Na^+))$ = 0.005, $u(w(Sr^{2+}))$ = 0.0078, $u(w(Mg^{2+}))$ = 0.003, $u(w(Br^-))$ = 0.003.

bottles containing salts and distilled water were placed in the thermostatic water bath oscillators (HZS−HA). The solution reached saturated solution through the machine oscillation. This process usually takes 15 days. When the concentrations of ions in the solutions remain unchanged, the solutions can attain equilibrium.

The wet residues solids were evaluated by chemical analysis. The components of the solid phase were identified by Schreinermarker wet residues or X-ray diffraction.

2.3. Analytical Methods.[17] The concentration of bromide ion (Br^-) was analyzed by the argentometry method. Under the conditions of coexistence of Sr^{2+} and Mg^{2+}, the total concentrations of Sr^{2+} and Mg^{2+} were obtained by EDTA titration; Eriochrome Black T was employed as an indicator. The strontium ion (Sr^{2+}) concentration was measured by EDTA standard solution; K−B indicator was employed as an indicator, and magnesium ion (Mg^{2+}) was precipitated down by adding sodium hydroxide solution. The concentration of Mg^{2+} was obtained by the total concentration minusing the concentration of Sr^{2+}. The concentration of potassium ion (K^+) was measured by a sodium tetraphenylborate (STPB)−hexadecyl trimethylammonium bromide (CTAB) titration. The concentration of sodium ion (Na^+) is obtained by the charge balance of ions.

3. RESULTS AND DISCUSSION

3.1. The $NaBr-SrBr_2-MgBr_2-H_2O$ System at 323 K. The solubility data, Jänecke dry-salt indices, and equilibrium solids of quaternary system $NaBr-SrBr_2-MgBr_2-H_2O$ at 323 K are listed in Table 2, with the solubility diagram shown in Figure 1. The solubility data are expressed in 100w(B). The Jänecke indices of NaBr, $SrBr_2$, $MgBr_2$, and H_2O can be calculated with the formulas below:

$$J(\mathrm{B}) = 100\frac{w(\mathrm{B})}{w(\mathrm{S})}$$

$$J(\mathrm{H_2O}) = 100\frac{w(\mathrm{H_2O})}{w(\mathrm{S})}$$

$$w(\mathrm{S}) = w(\mathrm{NaBr}) + w(\mathrm{SrBr_2}) + w(\mathrm{MgBr_2})$$

In the formulas, “S” represents “all dry salts”, and “B” represents $SrBr_2$ or $MgBr_2$, NaBr.

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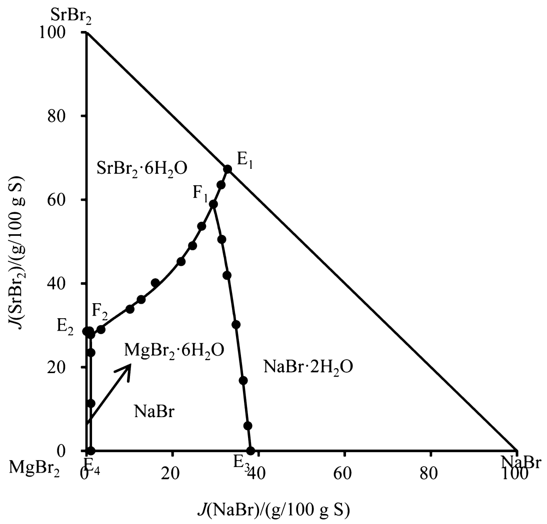

Figure 1. Dry-salt solubility diagram of quaternary system NaBr−$SrBr_2$−$MgBr_2$−H_2O at 323 K.

E_1, E_2, E_3, and E_4 are the invariant points of the ternary subsystems NaBr−$SrBr_2$−H_2O, $MgBr_2$−$SrBr_2$−H_2O, and NaBr−$MgBr_2$−H_2O, respectively. E_1F_1, F_1F_2, E_2F_2, E_4F_2, and E_3F_1 are the univariant curves consisting of two solids and the saturated solution.

Point F_1 is one of the invariant points, saturated with salts NaBr·$2H_2O$, SrBr·$6H_2O$, and NaBr, with the composition of liquid phase w(NaBr) = 16.96%, $w(MgBr_2)$ = 6.70%, and $w(SrBr_2)$ = 33.95%;

Point F_2 is another invariant point, saturated with salts $MgBr_2$·$6H_2O$, SrBr·$6H_2O$, and NaBr, with the composition of liquid phase w(NaBr) = 0.53%, $w(MgBr_2)$ = 39.41%, and $w(SrBr_2)$ = 15.35%.

There are four crystalline regions representing SrBr·$6H_2O$, NaBr, $MgBr_2$·$6H_2O$, and NaBr·$2H_2O$, respectively. The salt of NaBr·$2H_2O$ has a larger crystalline region, and the salt of $MgBr_2$·$6H_2O$ has the smallest crystalline region, whereas the solubility of NaBr·$2H_2O$ in the system is smaller and the solubility of $MgBr_2$·$6H_2O$ is the biggest.

Figure 2 shows that water content decreases with the increasing of the Jänecke indices of NaBr at the univariant curve

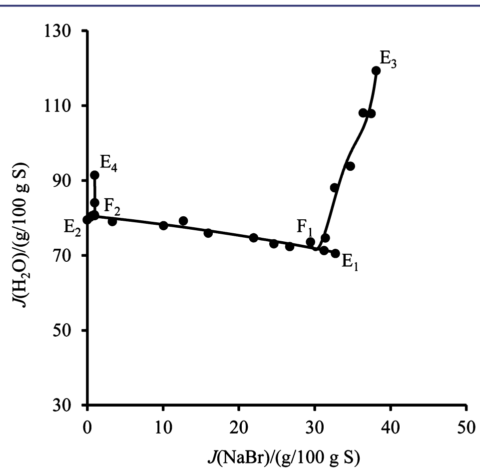

Figure 2. Water contents of saturated solutions in quaternary system NaBr−$SrBr_2$−$MgBr_2$−H_2O at 323 K.

F_2E_1. The water content reaches the biggest value 119.33g/100 g at the point E_3.

The X-ray diffraction photographs of the invariant points F_1 and F_2 in the quaternary system NaBr−$SrBr_2$−$MgBr_2$−H_2O at 323 K are given in Figure 3 and Figure 4.

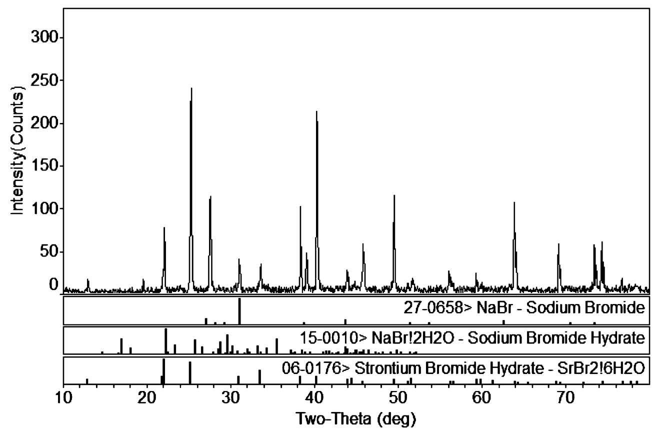

Figure 3. X-ray diffraction photograph of the invariant point F_1 of the quaternary system NaBr−$SrBr_2$−$MgBr_2$−H_2O at 323 K.

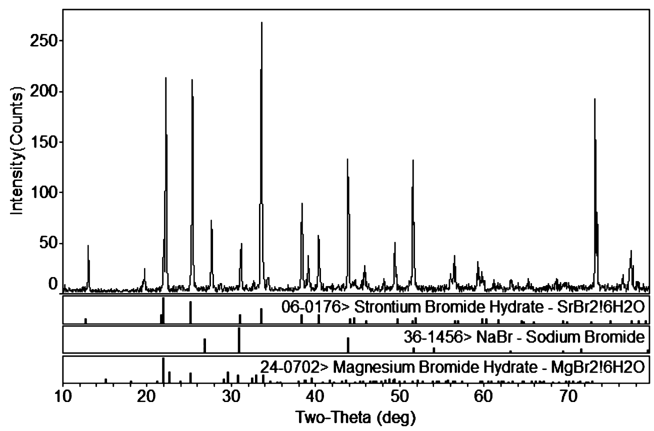

Figure 4. X-ray diffraction photograph of the invariant point F_2 of the quaternary system NaBr−$SrBr_2$−$MgBr_2$−H_2O at 323 K.

3.2. The KBr−$SrBr_2$−$MgBr_2$−H_2O System at 323 K.

The experimental results of solubilities in the quaternary system KBr−$SrBr_2$−$MgBr_2$−H_2O at 323 K were measured and are presented in Table 3. A dry-salt solubility diagram and a water content diagram are plotted in Figure 5 and Figure 6, respectively.

In Figure 5, the system has a double salt (KBr·$MgBr_2$·$6H_2O$) without solid solution formed. Point F_3 is one invariant point of the quaternary system, saturated with salts KBr + KBr·$MgBr_2$·$6H_2O$ + $SrBr_2$·$6H_2O$, which the composition of liquid phase is w(KBr) = 4.68%, $w(MgBr_2)$ = 16.71%, $w(SrBr_2)$ = 34.41%; point F_4 is another invariant point of the system, saturated with salts $MgBr_2$·$6H_2O$ + KBr·$MgBr_2$·$6H_2O$ + $SrBr_2$·$6H_2O$, which the composition of liquid phase is w(KBr) = 0.57%, $w(MgBr_2)$ = 36.30%, $w(SrBr_2)$ = 15.19%.

There are four crystalline regions representing potassium chloride (KBr), magnesium bromide hexahydrate ($MgBr_2$·$6H_2O$), strontium bromide hexahydrate ($SrBr_2$·$6H_2O$), and double salt (KBr·$MgBr_2$·$6H_2O$).

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Table 3. Experimental Values of Solubilities and Jänecke Indices of Dry Salt (J) of the Quaternary System $KBr-SrBr_2-MgBr_2-H_2O$ at 323 K and Pressure p = 0.1 MPa[a,b]

	composition of solution $w(B) \times 100$			Jänecke index J (g/100 g) $J(KBr) + J(MgBr_2) + J(SrBr_2)$ = 100 g				
no.	$w(KBr)$	$w(MgBr_2)$	$w(SrBr_2)$	$J(KBr)$	$J(MgBr_2)$	$J(SrBr_2)$	$J(H_2O)$	equilibrium solids
1, E_5	7.03	0.00	50.76	12.16	0.00	87.84	73.04	KB + SB
2	7.01	0.68	50.17	12.12	1.18	86.70	72.82	KB + SB
3	6.57	3.35	47.26	11.49	5.85	82.66	74.89	KB + SB
4	6.18	6.05	44.76	10.84	10.62	78.54	75.49	KB + SB
5	5.69	10.45	41.03	9.95	18.28	71.77	74.91	KB + SB
6	5.21	13.18	38.32	9.19	23.23	67.57	76.33	KB + SB
7	3.56	21.12	31.16	6.37	37.82	55.81	79.09	KMB + SB
8	2.30	25.96	24.75	4.34	48.97	46.69	88.62	KMB + SB
9	1.60	30.61	19.90	3.07	58.73	38.20	91.90	KMB + SB
10	0.89	34.86	16.46	1.70	66.77	31.53	91.54	KMB + SB
11, E_6	0.00	40.07	16.09	0.00	71.36	28.64	78.06	MB + SB
12	0.17	40.25	16.18	0.29	71.12	28.58	76.68	MB + SB
13	0.56	40.52	16.07	0.98	70.90	28.12	74.98	MB + SB
14, E_7	0.80	51.80	0.00	1.52	98.48	0.00	90.12	MB + KMB
15	0.80	49.79	2.63	1.50	93.57	4.94	87.93	MB + KMB
16	0.74	47.32	4.70	1.40	89.69	8.91	89.56	MB + KMB
17	0.76	45.48	8.84	1.38	82.58	16.04	81.56	MB + KMB
18, F_4	0.57	36.30	15.19	1.10	69.72	29.18	92.05	MB + KMB + SB
19, E_8	4.88	44.33	0.00	9.92	90.08	0.00	103.21	KB + KMB
20	4.88	40.54	5.53	9.58	79.56	10.86	96.26	KB + KMB
21	5.07	34.26	13.16	9.67	65.26	25.07	90.50	KB + KMB
22	4.91	29.34	21.77	8.76	52.38	38.86	78.50	KB + KMB
23	4.94	24.64	26.82	8.77	43.69	47.54	77.30	KB + KMB
24, F_3	4.68	16.71	34.41	8.39	29.95	61.67	79.22	KB + KMB + SB

[a]KB, KBr; KMB, $KBr\cdot MgBr_2\cdot 6H_2O$; MB, $MgBr_2\cdot 6H_2O$; SB, $SrBr_2\cdot 6H_2O$. [b]Standard uncertainties u are $u(T)$ = 0.1 K, $u_r(p)$ = 0.05, $u(w(K^+))$ = 0.003, $u(w(Sr^{2+}))$ = 0.0078, $u(w(Mg^{2+}))$ = 0.003, $u(w(Br^-))$ = 0.003.

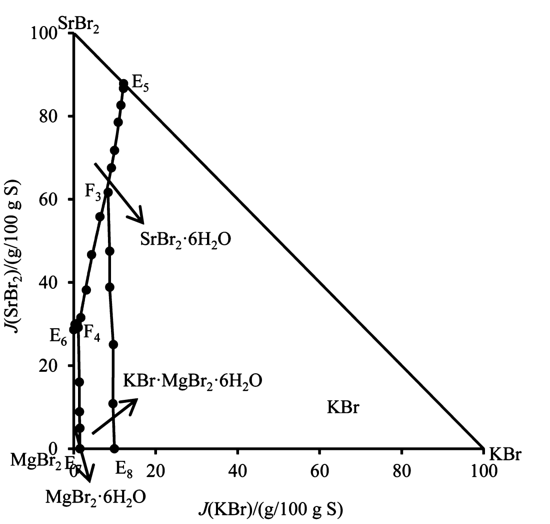

Figure 5. Dry-salt solubility diagram of the quaternary system $KBr-SrBr_2-MgBr_2-H_2O$ at 323 K.

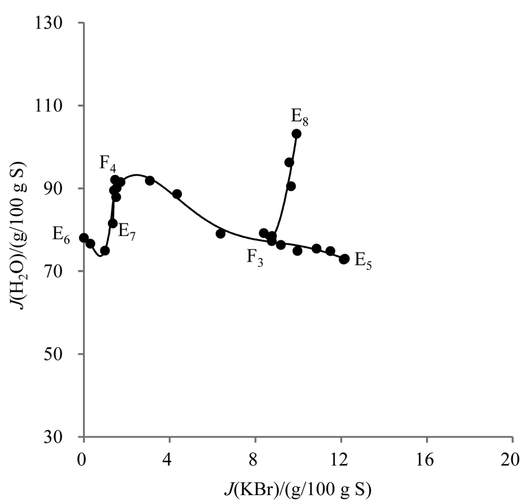

Figure 6. Water contents of saturated solutions in quaternary system $KBr-SrBr_2-MgBr_2-H_2O$ at 323 K.

The crystalline region of KBr is the largest in the system, which shows that the solubility of KBr is the smallest. The salt KBr can be crystallized out from the solution easily.

In Figure 6, it reveals that there has been a gradual decline at the univariant curve F_4E_5, while there has been a marked increase at the univariant curve F_3E_8. The water content reaches the biggest value 103.21g/100 g at the point E_8.

Figure 7 and Figure 8 are X-ray diffraction photographs of points at univariant curves E_5F_3 and E_6F_4 of system $KBr-SrBr_2-MgBr_2-H_2O$ at 323 K, respectively.

4. CONCLUSIONS

The system $NaBr-SrBr_2-MgBr_2-H_2O$ at 323 K has four solid phase crystallization areas corresponding to strontium bromide hexahydrate ($SrBr_2\cdot 6H_2O$), magnesium bromide hexahydrate ($MgBr_2\cdot 6H_2O$), sodium bromide (NaBr), and sodium bromide

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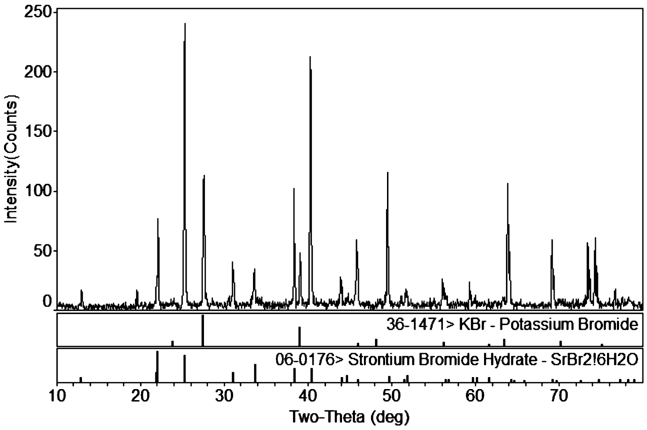

Figure 7. X-ray diffraction photograph of a point on the curve E_5F_3 of the quaternary system $KBr-SrBr_2-MgBr_2-H_2O$ at 323 K.

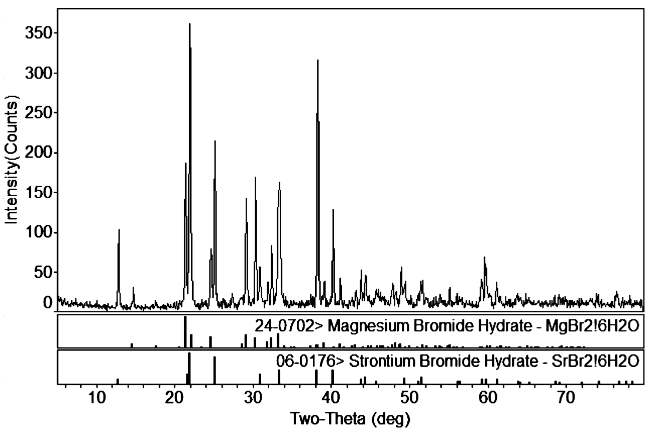

Figure 8. X-ray diffraction photograph of a point on the curve E_6F_4 of the quaternary system $KBr-SrBr_2-MgBr_2-H_2O$ at 323 K.

dihydrate ($NaBr·2H_2O$). The system $KBr-SrBr_2-MgBr_2-H_2O$ at 323 K has a double salt ($KBr·MgBr_2·6H_2O$) formed. The system at 323 K has four solid phase crystallization areas corresponding to strontium bromide hexahydrate ($SrBr_2·6H_2O$), magnesium bromide hexahydrate ($MgBr_2·6H_2O$), complex salt ($KBr·MgBr_2·6H_2O$), and potassium bromide (KBr); both of them have two invariant points and five univariant curves.

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Notes

The authors declare no competing financial interest.

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