Solid–Liquid Equilibria in Ternary System $SrBr_2$–$MgBr_2$–H_2O at (298 and 323) K

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In this study, solid–liquid equilibrium measurements in ternary system $SrBr_2$–$MgBr_2$–H_2O at (298 and 323) K are conducted using the isothermal dissolution equilibrium method. The mass fraction of every component of the equilibrium liquid phase in the system is determined at (298 and 323) K. Based on the experimental results, the phase diagrams and density-composition diagrams are plotted. Results show that no double salt or solid solution is formed in the system $SrBr_2$–$MgBr_2$–H_2O at (298 and 323) K. The ternary system $SrBr_2$–$MgBr_2$–H_2O at (298 and 323) K has one invariant point, two univariant curves, and there are two solid–liquid two-phase areas that correspond to strontium bromide hexahydrate ($SrBr_2·6H_2O$) and magnesium bromide hexahydrate ($MgBr_2·6H_2O$).

Introduction

With the continuous development of modern industries, there has been significant exploitation of mineral resources, and the exploitation and utilization of underground brines have become increasingly important. China possesses rich underground brine resources. The underground brines of Sichuan basin are a vast treasure house of liquid mineral resources, and have many useful components, such as K^+, Br^-, I^-, Li^+, Sr^{2+}, and Rb^+ (Lin, 2006). Its products of bromine, iodine, potassium chloride, barium chloride, strontium carbonate, lithium carbonate, and rubidium chloride are the raw materials of chemical, military, nuclear, aerospace, and electronic industries, as well as other high-tech fields (Lin and Cao, 1998). While these products are in short supply, they remain in high demand, and have vast market prospects that are very competitive. The appropriate development and use of these resources will not only promote the development of the halogenated solvent industry in Sichuan, but also greatly mitigate the present shortage of resources in China (Lin and Chen, 2008). Research into the phase equilibrium of salt water systems can provide theoretical support for the comprehensive exploitation and utilization of salt-lake brine resources.

Up to the present, a series of studies have been conducted on the phase equilibria of systems containing strontium or bromide, such as the Na–K–Sr–Cl–H_2O system and its sub-systems Na–Sr–Cl–H_2O and K–Sr–Cl–H_2O at (291, 323, and 373) K (Assarsson, 1953), as well as Na–K–Sr–Ca–Cl–H_2O systems at (291, 309.5, and 373) K (Assarsson and Balder, 1954a). Further, ternary systems Na–K–Br–H_2O and K–Mg–Br–H_2O at 323 K (Christov, 2007, 2011), Mg–Sr–Cl–H_2O at (292, 353 and 373) K (Assarsson and Balder, 1954b), and multicomponent systems Na–K–Sr–Ca–Mg–Cl–H_2O at (295 and 366) K (Assarsson and Balder, 1955) have been studied.

In addition, our group has performed many related phase-equilibrium measurements for systems containing Sr^{2+} or Br^-, for example, the ternary systems Na–Sr–Cl–H_2O and K–Sr–Cl–H_2O at 348 K (Li *et al.*, 2015a), Mg–Sr–Cl–H_2O at (323 and 348 K) (Li *et al.*, 2015b), $K_2B_4O_7$–KBr–H_2O and $Na_2B_4O_7$–NaBr–H_2O at 298 K (Sang *et al.*, 2006; Sang and Yu., 2006), KBr–$CaBr_2$–H_2O and NaBr–$CaBr_2$–H_2O at 348 K (Hu *et al.*, 2015). The phase equilibria of the quaternary systems $Na_2B_4O_7$–NaBr–Na_2SO_4–H_2O, NaCl–NaBr–$Na_2B_4O_7$–H_2O at 348 K (Ning *et al.*, 2012; Li *et al.*, 2013), Na^+, K^+//Br^-, $B_4O_7^{2-}$–H_2O at 298 K (Cui *et al.*, 2014), Na^+, K^+//Br^-, SO_4^{2-}–H_2O and KCl–KBr–K_2SO_4−H_2O at 323 K (Sang *et al.*, 2011; Wang *et al.*, 2011), and the quinary system KCl–KBr–K_2SO_4–$K_2B_4O_7$–H_2O at (323 and 348) K (Cui *et al.*, 2013) have been reported.

To the best of our knowledge, there are no reports on the solid–liquid equilibria of the ternary system $SrBr_2$–$MgBr_2$–H_2O at (298 and 323) K. Therefore, the purpose of this work is to measure the phase equilibria of the above ternary system at (298 and 323) K. The experiment content has a specific value for the development and utilization of underground brine containing strontium.

1. Experimental

1.1 Reagents and instruments

The chemical reagent used in this experiment was analytical reagent: $SrBr_2·6H_2O$, 99% pure (produced by Shanghai Haorui Chemical Limited Corporation); $MgBr_2·6H_2O$, 98%

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pure (produced by Shanghai Xinbao Fine Chemical Plant); the distilled water used in the experiment was produced using a water-polishing system. When the conductivity was lower than 1×10^{-5} S·m^{-1} and pH = 6, the water reached the use standard.

A standard analytical balance (AL104) with a 110-g capacity produced by the Mettler Toledo Instruments Co., Ltd. was employed to weigh the mass of the solution.

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

1.2 Experimental methods

The experiments here were conducted using the isothermal solution equilibrium method.

1. Preparation of saturated solution of the first salt and water at 298 or 323 K.
2. Removal of solid of the first salt from the solutions.
3. Addition of different amounts of the second salt in each binary solution.
4. Equilibration

The chemical agents and distilled water were mixed in 150-mL plastic bottles. Then, all the bottles were placed in the thermostatic water bath oscillators (HZS–HA) in order. The solution became saturated after the machine oscillation. This process usually took 15 d (Niu and Cheng, 2002). The liquid-phase compositions of the solution were analyzed once every few days. When the concentrations of ions in the solution remain unchanged, the solution can reach equilibrium.

The components of the solids phase were identified by X-ray diffraction. The densities were measured using the pycnometer method with a density bottle, for which the precision is 0.0002 g·cm^{-3}. Before the determination, the pure water was used at 298 K or 323 K to correct the density bottle and analytical balance respectively, which then achieves the corresponding correction coefficient.

1.3 Analytical methods

The concentration of bromide ion (Br^-) was measured using the argentometry method. Under the condition of co-existence of Sr^{2+} and Mg^{2+}, the magnesium ion (Mg^{2+}) was precipitated down by adding sodium hydroxide solution. The strontium ion concentration (Sr^{2+}) was measured using EDTA titration, and a K–B indicator (acid chrome blue K/naphtha green B = 1 : 2) was employed as an indicator. The (1+1) hydrochloric acid was added to the above solution, which was used to neutralize the above alkaline solution in the continuous titration process. Then, the concentration of magnesium ion (Mg^{2+}) was obtained by EDTA titration in the presence of a buffer solution, and the K–B indicator was employed as an indicator.

2. Results and Discussion

2.1 $SrBr_2$–$MgBr_2$–H_2O system at 298 K

The solubility data, solution density, and equilibrium solids of the ternary system $SrBr_2$–$MgBr_2$–H_2O at 298 K are listed in **Table 1**. The solubility data were expressed in $100\cdot w$ (B), and w (B) denotes the mass fraction of salt.

The phase diagram of ternary system $SrBr_2$–$MgBr_2$–H_2O at 298 K is shown in **Figure 1**, and the density–composition diagram of the system at 298 K is presented in **Figure 2**.

The univariant curve BE shows the addition of the salt of $MgBr_2$ into the saturated solution of $SrBr_2$ and H_2O, and conversely, $SrBr_2$ was added to the $MgBr_2$ solution for the curve AE. Point E is the invariant point of the ter-

Table 1 Solubility and density values of solution in the ternary system $SrBr_2$–$MgBr_2$–H_2O at 298 K and pressure p = 0.1 MPa

No	Composition of liquid phase, $100\cdot w$		Solution density ρ [g·cm^{-3}]	Equilibrium solid phase
	$SrBr_2$	$MgBr_2$		
1,A	0.00	50.62	1.5939	$MgBr_2\cdot6H_2O$
2	0.17	50.16	1.5968	$MgBr_2\cdot6H_2O$
3	0.89	49.89	1.6077	$MgBr_2\cdot6H_2O$
4	1.45	48.97	1.6098	$MgBr_2\cdot6H_2O$
5	2.09	48.40	1.6136	$MgBr_2\cdot6H_2O$
6	2.74	48.04	1.6172	$MgBr_2\cdot6H_2O$
7	3.25	47.94	1.6199	$MgBr_2\cdot6H_2O$
8	4.02	47.26	1.6251	$MgBr_2\cdot6H_2O$
9	4.92	46.98	1.6298	$MgBr_2\cdot6H_2O$
10,E	5.31	46.65	1.6335	$MgBr_2\cdot6H_2O+SrBr_2\cdot6H_2O$
11	9.86	40.85	1.6352	$SrBr_2\cdot6H_2O$
12	13.10	33.37	1.6378	$SrBr_2\cdot6H_2O$
13	18.37	27.13	1.6404	$SrBr_2\cdot6H_2O$
14	24.53	20.93	1.6418	$SrBr_2\cdot6H_2O$
15	32.17	13.66	1.6484	$SrBr_2\cdot6H_2O$
16	38.10	8.47	1.6529	$SrBr_2\cdot6H_2O$
17	43.10	4.34	1.6571	$SrBr_2\cdot6H_2O$
18	46.80	0.97	1.6614	$SrBr_2\cdot6H_2O$
19,B	51.70	0.00	1.6701	$SrBr_2\cdot6H_2O$

Standard uncertainties (u) are $u(T)$ = 0.1 K, $u(p)$ = 0.05 MPa, $u(w(Sr^{2+}))$ = 0.0078, $u(w(Mg^{2+}))$ = 0.003, $u(w(Br^-))$ = 0.003

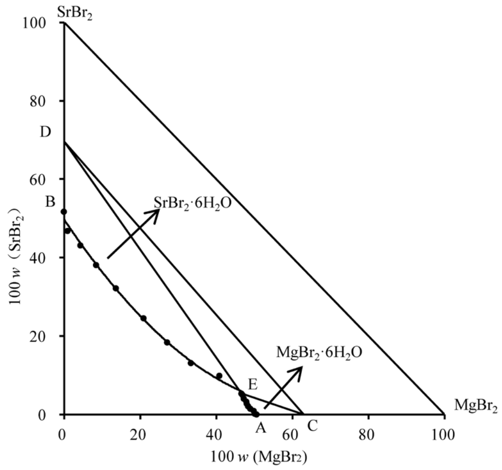

Fig. 1 Phase diagram of ternary system $SrBr_2$–$MgBr_2$–H_2O at 298 K

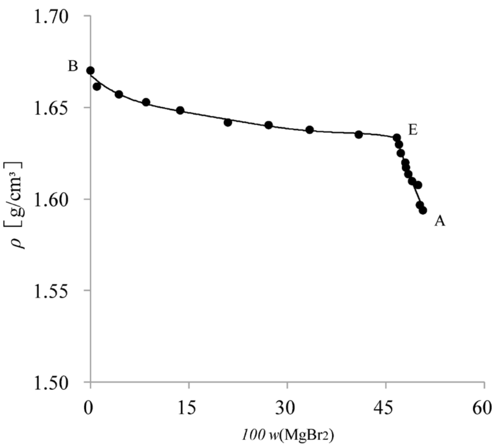

Fig. 2 Density-composition diagram of ternary system $SrBr_2$–$MgBr_2$–H_2O at 298 K

nary system, saturated with salts $MgBr_2$ and $SrBr_2$, with the composition of liquid phase w ($MgBr_2$) = 46.65%, w ($SrBr_2$) = 5.31%.

There are two solid–liquid two-phase regions that represent $SrBr_2 \cdot 6H_2O$ and $MgBr_2 \cdot 6H_2O$. The solid–liquid two-phase regions of $MgBr_2 \cdot 6H_2O$ are smaller than that of $SrBr_2 \cdot 6H_2O$, showing that the solubility of $MgBr_2 \cdot 6H_2O$ in the system is greater. $SrBr_2 \cdot 6H_2O$ can be extracted from the solution easily.

Figure 2 shows the density change with w ($MgBr_2$). There is a slight decline in the density of the solution on the curve BE. The density decreases with a marked increasing of w ($MgBr_2$) on the curve AE. The density reaches its maximum value of 1.6701 $g \cdot cm^{-3}$ at the boundary point B.

The solid–phase composition was determined using X-ray diffraction. **Figure 3** is an X-ray diffraction photograph of a point on univariant curve BE of the ternary system $SrBr_2$–$MgBr_2$–H_2O at 298 K. Figure 3 shows that the equilibrium solid-phase composition of $SrBr_2$ is $SrBr_2 \cdot 6H_2O$. The X-ray diffraction photograph of the invariant point E in the ternary system at 298 K is given in **Figure 4**. According to this figure, point E corresponds to the saturation point of

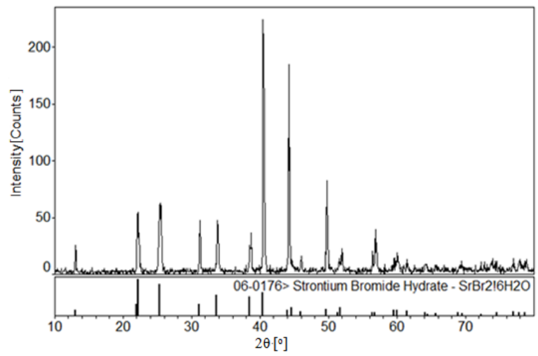

Fig. 3 X-ray diffraction photograph of a point on the curve BE of the ternary system $SrBr_2$–$MgBr_2$–H_2O at 298 K

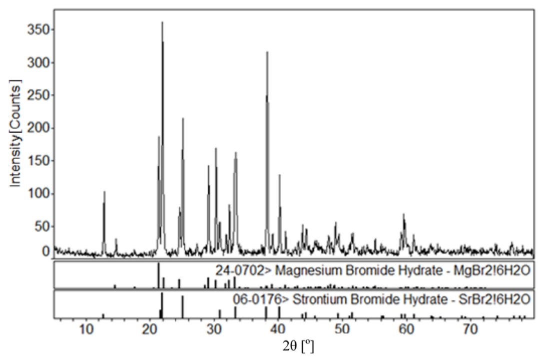

Fig. 4 X-ray diffraction photograph of the invariant point E of the ternary system $SrBr_2$–$MgBr_2$–H_2O at 298 K

$SrBr_2 \cdot 6H_2O$ and $MgBr_2 \cdot 6H_2O$.

2.2 The $SrBr_2$–$MgBr_2$–H_2O system at 323 K

The experimental results of the solubility and density values obtained for the ternary system $SrBr_2$–$MgBr_2$–H_2O at 323 K are presented in **Table 2**. Using the experimental data, the equilibrium phase diagram of this system at 323 K is as plotted in **Figure 5**. The density–composition diagram of the system at 323 K is presented in **Figure 6**.

In Figure 5, Point A_1 is the invariant point of $MgBr_2$–H_2O and B_1 is the invariant point of $SrBr_2$–H_2O at 323 K. There were two univariant curves that correspond to curves A_1E_1 and B_1E_1. These curves represent the boundary of the single-liquid phase region and solid–liquid two-phase region. Point E_1 is the invariant point of $SrBr_2$–$MgBr_2$–H_2O at 323 K, and it was saturated with two salts: $MgBr_2 \cdot 6H_2O$ and $SrBr_2 \cdot 6H_2O$, the composition of the liquid phase is w($MgBr_2$) = 39.81%, w($SrBr_2$) = 15.94%. There are two solid–liquid two-phase regions for the solid $SrBr_2 \cdot 6H_2O$ and $MgBr_2 \cdot 6H_2O$. The solid–liquid two-phase region of $SrBr_2 \cdot 6H_2O$ is larger, while the solid–liquid two-phase region of $MgBr_2 \cdot 6H_2O$ is smaller. It shows that $MgBr_2$ has a larger solubility than $SrBr_2$.

The density data are shown in Figure 6. From the figure, we can understand the density trend with w ($MgBr_2$). The density declines with the increasing content of $MgBr_2$ on the curve B_1A_1. At point A_1, the density achieves a minimum

Table 2 Solubility and density values of solution in the ternary system $SrBr_2$–$MgBr_2$–H_2O at 323 K and pressure $p = 0.1$ MPa

No	Composition of liquid phase, 100w		Solution density, ρ[g·cm^{-3}]	Equilibrium solid phase
	$SrBr_2$	$MgBr_2$		
1,A_1	0.00	52.17	1.6273	$MgBr_2 \cdot 6H_2O$
2	0.96	51.10	1.6285	$MgBr_2 \cdot 6H_2O$
3	2.05	49.26	1.6345	$MgBr_2 \cdot 6H_2O$
4	4.35	46.25	1.6414	$MgBr_2 \cdot 6H_2O$
5	8.34	43.10	1.6559	$MgBr_2 \cdot 6H_2O$
6	9.50	42.42	1.6590	$MgBr_2 \cdot 6H_2O$
7	10.30	41.98	1.6649	$MgBr_2 \cdot 6H_2O$
8	13.48	40.35	1.6752	$MgBr_2 \cdot 6H_2O$
9,E_1	15.94	39.81	1.6822	$MgBr_2 \cdot 6H_2O + SrBr_2 \cdot 6H_2O$
10	18.28	34.12	1.6934	$SrBr_2 \cdot 6H_2O$
11	20.54	30.66	1.7011	$SrBr_2 \cdot 6H_2O$
12	26.55	23.36	1.7110	$SrBr_2 \cdot 6H_2O$
13	36.04	14.58	1.7284	$SrBr_2 \cdot 6H_2O$
14	48.19	5.67	1.7472	$SrBr_2 \cdot 6H_2O$
15	53.36	2.36	1.7574	$SrBr_2 \cdot 6H_2O$
16,B_1	57.60	0.00	1.7690	$SrBr_2 \cdot 6H_2O$

Standard uncertainties (u) are $u(T) = 0.1$ K, $u_r(p) = 0.05$ MPa, $u(w(Sr^{2+})) = 0.0078$, $u(w(Mg^{2+})) = 0.003$, $u(w(Br^-)) = 0.003$

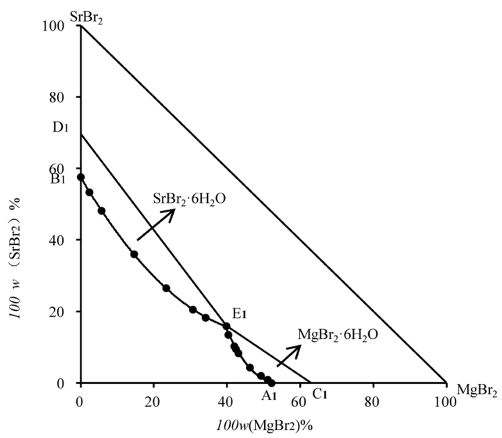

Fig. 5 Phase diagram of ternary system $SrBr_2$–$MgBr_2$–H_2O at 323 K

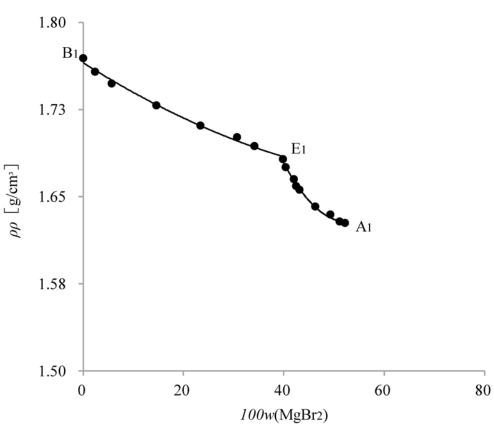

Fig. 6 Density–composition diagram of ternary system $SrBr_2$–$MgBr_2$–H_2O at 323 K

value of 1.6273 g·cm^{-3}.

Figure 7 is an X-ray diffraction photograph of a point on univariant curve A_1E_1 of the ternary system $SrBr_2$–$MgBr_2$–H_2O at 323 K. From Figure 7, the equilibrium solid-phase composition of $MgBr_2$ is $MgBr_2 \cdot 6H_2O$. **Figure 8** is the X-ray diffraction photograph of the invariant point E_1 in the ternary system $SrBr_2$–$MgBr_2$–H_2O at 323 K. According to Figure 8, point E_1 corresponds to the saturation point of

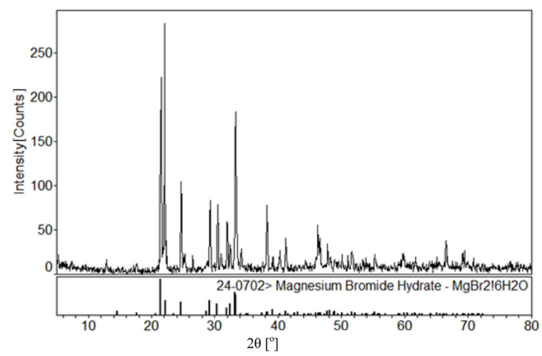

Fig. 7 X-ray diffraction photograph of a point on the curve A_1E_1 of the ternary system $SrBr_2$–$MgBr_2$–H_2O at 323 K

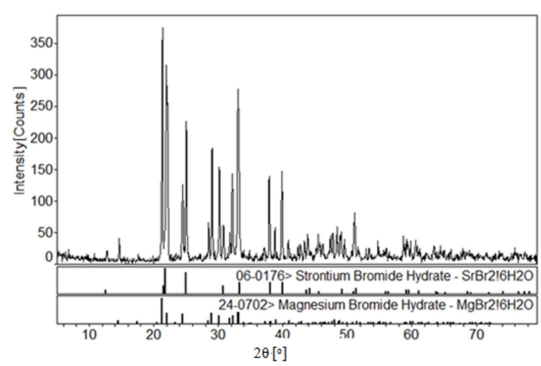

Fig. 8 X-ray diffraction photograph of the invariant point E_1 of the ternary system $SrBr_2$–$MgBr_2$–H_2O at 323 K

$SrBr_2 \cdot 6H_2O$ and $MgBr_2 \cdot 6H_2O$.

2.3 Comparison of results of ternary system $SrBr_2$–$MgBr_2$–H_2O at (298 and 323) K

Comparing Figures 1 and 5, the ternary system $SrBr_2$–$MgBr_2$–H_2O has one invariant point, two univariant curves, and two solid–liquid two-phase regions that correspond to $SrBr_2 \cdot 6H_2O$ and $MgBr_2 \cdot 6H_2O$ at both (298 and 323) K. The solid-liquid two–phase regions of salt $MgBr_2 \cdot 6H_2O$ are both smaller than that of salt $SrBr_2 \cdot 6H_2O$; therefore $MgBr_2$ has a larger solubility than $SrBr_2$ in the ternary system at (298 and 323) K. As the temperature rises, the solubility curves of the system tend towards an increased total salinity, and the concentration of liquid salts increases. The crystallization field of $SrBr_2 \cdot 6H_2O$ becomes smaller because of the rapid increase in the solubility of $SrBr_2 \cdot 6H_2O$ with temperature.

Conclusions

The phase equilibrium in ternary system $SrBr_2$–$MgBr_2$–H_2O at (298 and 323) K was determined using the isothermal dissolution equilibrium method. The mass fraction of every component of the equilibrium liquid phase in the system at (298 and 323) K was determined. Based on the experimental results, the phase diagrams and density composition diagrams were plotted. The system $SrBr_2$–$MgBr_2$–H_2O at (298 and 323) K has one invariant point, two univariant curves, and two solid–liquid two-phase regions, which correspond to $SrBr_2 \cdot 6H_2O$ and $MgBr_2 \cdot 6H_2O$. In addition, the salt of $SrBr_2 \cdot 6H_2O$ has a larger solid–liquid two-phase region than the salt of $MgBr_2 \cdot 6H_2O$.

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