Research Article

Study of Phase Equilibrium of $\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$ and $\text{NaBr} + \text{MgBr}_2 + \text{H}_2\text{O}$ at 313.15 K

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The phase equilibrium for the ternary systems $\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$ and $\text{NaBr} + \text{MgBr}_2 + \text{H}_2\text{O}$ at 313.15 K was investigated by isothermal solution saturation method. The solubilities of salts and the densities of saturated solutions in these ternary systems were determined by chemical methods, while the equilibrium solid phases were analyzed by Schreinermarker wet residues method. Based on the experimental data, phase diagrams and density versus composition diagrams were plotted. The two ternary systems were type of simple common-saturation and without complex salt and solid solution. There are in all two crystalline regions, two univariant curves, and one invariant point in these phase diagrams of two ternary systems at 313.15 K. The equilibrium solid phases in the ternary system $\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$ are KBr and $\text{NaBr} \cdot 2\text{H}_2\text{O}$, and those in the ternary system $\text{NaBr} + \text{MgBr}_2 + \text{H}_2\text{O}$ are $\text{NaBr} \cdot 2\text{H}_2\text{O}$ and $\text{MgBr}_2 \cdot 6\text{H}_2\text{O}$.

1. Introduction

Phase equilibrium in salt-water systems and phase diagram are the foundation of inorganic chemical production and salt mineral resources exploitation [1–4]. To extract relevant products from the potassium, magnesium, and bromine salt mine, it is essential to investigate the phase equilibrium of $\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$ and $\text{NaBr} + \text{MgBr}_2 + \text{H}_2\text{O}$. By now, a number of studies on the Br-bearing phase equilibria have been done, such as quaternary systems $\text{KCl}-\text{KBr}-\text{K}_2\text{SO}_4-\text{H}_2\text{O}$ at 323 K, 348 K, and 373 K [5–7], $\text{NaBr}-\text{SrBr}_2-\text{MgBr}_2-\text{H}_2\text{O}$ and $\text{KBr}-\text{SrBr}_2-\text{MgBr}_2-\text{H}_2\text{O}$ at 323 K [8], and quinary system Na^+ , $\text{K}^+//\text{Cl}^-$, Br^- , and $\text{SO}_4^{2-}-\text{H}_2\text{O}$ at 373 K [9]. The two ternary systems $\text{NaBr}-\text{KBr}-\text{H}_2\text{O}$ and $\text{NaBr}-\text{MgBr}_2-\text{H}_2\text{O}$ also have been reported at 323 K and 348 K [10–12]. However, the data provided is far from enough, so an extensive study at other temperatures needs to be done. The phase equilibrium of $\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$ and $\text{NaBr} + \text{MgBr}_2 + \text{H}_2\text{O}$ at 313.15 K has not been reported yet. This paper is conducive to fill the blank of data. In this study, the solubility and density of the ternary systems were obtained. The equilibrium solid phases were analyzed, and the crystallization regions were determined. All results can offer fundamental data support

for salt mineral resources exploitation and further theoretical studies.

2. Methodology

2.1. Materials and Apparatus. The sources and purity of the chemicals are listed in Table 1. Doubly deionized water (electrical conductivity $\leq 1 \cdot 10^{-4} \text{ S} \cdot \text{m}^{-1}$) is used in the work. A HZS-H thermostatic water bath shaker is employed to carry out the experiments.

2.2. Experimental Methods. The method of isothermal solution saturation [13–15] was employed to determine the solubility of the ternary systems. The famous Schreinermarks method of moist residues [15–17] was applied to determine the equilibrium solid phase in the experiments.

Based on a fixed ratio and ensuring that one of the components is excessive, the experimental components are added to a series of conical flasks (125 mL) gradually, and the sealed flasks are placed into the oscillator. The oscillator vibrates continuously at 313.15 K (the standard uncertainty of 0.3 K). In a pre-experiment, the liquid phase of the samples

TABLE 1: Purities and suppliers of chemicals.

Chemical	Mass fraction purity	Source
NaBr	$\geq 99.0\%$	Tianjin Bodi Chemical Holding Co. Ltd., China
KBr	$\geq 99.0\%$	Tianjin Bodi Chemical Holding Co. Ltd., China
$\text{MgBr}_2 \cdot 6\text{H}_2\text{O}$	$\geq 99.0\%$	Tianjin Bodi Chemical Holding Co. Ltd., China

is analyzed every 2 days, and it is shown that the phase equilibrium is reached in 10 days. After equilibrium, the oscillation is stopped and the system is allowed to stand for 4 days to make sure that all the suspended crystals settle. The wet residues and liquid phase are transferred to two volumetric flasks, respectively. Simultaneously, some other liquid phases are used to determine density individually. Finally, these samples are quantitatively analyzed by chemical methods.

More details of the experimental method and the procedure are presented in the previous papers [12–14].

2.3. Analysis. The concentration of potassium ion was analyzed by a sodium tetraphenylborate (STPB) hexadecyl trimethyl ammonium bromide (CTAB) titration [18–20] (uncertainty of 0.0058); the concentration of magnesium ion was measured with an EDTA standard solution using the indicator Eriochrome Black-T [21] (uncertainty of 0.0072); the concentration of bromine ion was determined by Mohr's method using a silver nitrate standard solution [21] (uncertainty of 0.0037); and the concentration of sodium was evaluated according to the ion charge balance. The density is measured using a pycnometer (uncertainty of 0.002). Each experimental result is achieved from the average value of three parallel measurements.

3. Results and Discussion

To compare with literature data [22, 23], the experimental data on the solubility for NaBr, KBr, or MgBr_2 in pure water at 313.15 K are in good agreement with the literature values, which demonstrates that the experimental devices and methods are feasible.

3.1. Solid-Liquid Phase Equilibrium for $\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$.

The experimental data were listed in Table 2. The ion concentration values were expressed in mass fraction in the equilibrium solution. The solution densities were given in grams per cubic centimeter. According to the experimental results, the phase diagram was plotted in Figure 1 and the relationship of the solution densities was plotted in Figure 2. In the ternary system $\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$ at 313.15 K, it contains one invariant point, two univariant curves, and two crystallization regions.

As indicated in Figure 1, A, B, C, and W denote solid NaBr, solid KBr, solid $\text{NaBr} \cdot 2\text{H}_2\text{O}$, and H_2O , respectively; point S, an invariant point, reflects the cosaturated solution

FIGURE 1: Equilibrium phase diagram of the ternary system $\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$ at 313.15 K. ●, equilibrium liquid phase composition; ■, moist solid phase composition; A, pure solid of NaBr; B, pure solid of KBr; C, pure solid of $\text{NaBr} \cdot 2\text{H}_2\text{O}$; W, water; H, solubility of NaBr in water; P, solubility of KBr in water; S, cosaturated point of $\text{NaBr} \cdot 2\text{H}_2\text{O}$ and KBr.

FIGURE 2: Density versus $100w(\text{NaBr})$ in the ternary system ($\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$). H, S, and P have the same meaning as described in Figure 1.

of KBr and $\text{NaBr} \cdot 2\text{H}_2\text{O}$ at 313.15 K, with $w(\text{NaBr}) = 0.4612$ and $w(\text{KBr}) = 0.0820$; P and H denote the solubility of KBr and NaBr in water at 313.15 K, respectively. Two univariant solubility curves of this ternary system are PS and HS. Curve PS corresponds to the saturated KBr solution and visualizes changes of the KBr concentration with increasing the NaBr

TABLE 2: Mass Fraction Solubility of the ternary NaBr + KBr + H₂O system at temperature = 313.15 K and pressure = 0.1 MPa^a.

Number	Composition of liquid phase, 100w		Composition of wet residue phase, 100w		Densities of liquid phase	Equilibrium solid phase
	100w ₁ ^b	100w ₂	100w ₁	100w ₂	$\rho/(\text{g}\cdot\text{cm}^{-3})$	
1, P	0.00	43.51	ND ^c	ND	1.4208	KBr
2	3.56	40.23	2.45	59.74	1.4247	KBr
3	7.23	36.77	5.43	53.13	1.4286	KBr
4	10.88	33.61	6.84	58.31	1.4351	KBr
5	14.88	30.33	9.60	55.30	1.4446	KBr
6	18.75	27.21	10.57	59.18	1.4546	KBr
7	21.68	24.98	12.22	57.81	1.4636	KBr
8	25.23	22.18	14.66	54.92	1.4738	KBr
9	29.28	19.08	15.63	56.89	1.4863	KBr
10	33.05	16.62	15.95	59.95	1.5034	KBr
11	37.45	13.87	18.97	56.55	1.5254	KBr
12	41.23	11.48	18.92	59.52	1.5439	KBr
13, S	46.12	8.20	49.09	20.61	1.5658	NaBr·2H ₂ O + KBr
14	47.31	4.97	59.53	2.84	1.5462	NaBr·2H ₂ O
15	49.56	2.36	54.95	1.91	1.5355	NaBr·2H ₂ O
16, H	51.43	0.00	ND	ND	1.5296	NaBr·2H ₂ O

^aStandard uncertainties $u(T) = 0.3$ K, $u_r(p) = 0.05$, $u_r(K^+) = 0.0058$, $u_r(\text{Br}^-) = 0.0037$, and $u_r(\rho) = 0.002$. ^b w_1 , mass fraction of NaBr; w_2 , mass fraction of KBr. ^cND, not determined. H, S, and P have the same meaning as described in Figure 2.

concentration. Curve SH corresponds to the saturated NaBr solution and indicates changes of the NaBr concentration with the KBr concentration increasing in the equilibrating solution. The KBr concentration decreases sharply with increasing the NaBr concentration, which illustrates that NaBr has a strong salting-out effect on KBr.

As indicated in Figure 1, along the curve PS, we connect the composition points of wet residue phase with liquid phase and then extend the intersection of these straight lines which is approximately the equilibrium solid phase for KBr. The same method is utilized to analyze the equilibrium solid phase of SH, and the intersection is NaBr·2H₂O. WPSH denotes unsaturated region at 313.15 K. BPS denotes crystallization region of KBr, while SHC denotes crystallization region of NaBr·2H₂O. Zone BSC represents the mixed crystalline region of KBr + NaBr·2H₂O. It is obvious that the crystalline region of NaBr·2H₂O is much smaller than that of KBr.

The phase diagrams of the ternary system NaBr + KBr + H₂O at 323 and 348 K have been reported [10]. Apparently, the three phase diagrams have very similar shapes, each of them having an invariant point, two univariant curves, and two crystallization regions. The equilibrium solid phases in the ternary system NaBr + KBr + H₂O are potassium bromide (KBr) and sodium bromide dihydrate (NaBr·2H₂O) at 313 K and 323 K, and those are potassium bromide (KBr) and sodium bromide (NaBr) at 348 K.

Figure 2 indicates the relationship between the mass fraction of NaBr and the density in the solution. With increasing the NaBr concentration, the density first increases

and then the density declines afterwards. At the invariant point S, the density reaches a maximum value.

3.2. Solid-Liquid Phase Equilibrium for NaBr + MgBr₂ + H₂O. The phase equilibrium experimental data is shown in Table 3, and the ternary phase diagram is drawn in Figure 3.

As indicated in Figure 3, A, M, D, C, and W denote solid NaBr, solid MgBr₂·6H₂O, solid MgBr₂, solid NaBr·2H₂O, and H₂O, respectively; point Q, an invariant point, reflects the cosaturated solution of MgBr₂·6H₂O and NaBr·2H₂O at 313.15 K, with $w(\text{NaBr}) = 0.0418$ and $w(\text{MgBr}_2) = 0.4781$; N and H represent the solubility of MgBr₂ and NaBr in water at 313.15 K, respectively. Two univariant solubility curves of this ternary system are PS and HS. Curve NQ corresponds to the saturated MgBr₂ solution and visualizes changes of the MgBr₂ concentration with increasing the NaBr concentration. Curve QH corresponds to the saturated NaBr solution and indicates changes of the NaBr concentration with increasing the MgBr₂ concentration. The solubility of NaBr decreases sharply with increasing the MgBr₂ concentration.

The polarization of ions has a certain effect on the dissolution of ionic crystals. The results show that the ionic dipole intensity in the solution depends on the electric field strength. In this study, the electrolyte concentration increased with the higher solubility of MgBr₂ added to the solution; also, the polarity of the solution increases, and the dielectric coefficient of the dielectric medium is reduced, while the ionic electric field strength increases, making it easy to bound more water to its surrounding, so that the reduction of water in the dissolution of other substances leads to enhanced

TABLE 3: Mass Fraction Solubility of the ternary NaBr + MgBr₂ + H₂O system at temperature = 313.15 K and pressure = 0.1 MPa^a.

Number	Composition of liquid phase, 100w		Composition of wet residue phase, 100w		Densities of liquid phase $\rho/(\text{g}\cdot\text{cm}^{-3})$	Equilibrium solid phase
	100w ₁ ^b	100w ₂	100w ₁	100w ₂		
1, H	51.43	0.00	ND ^c	ND	1.5296	NaBr·2H ₂ O
2	45.58	4.98	59.07	2.67	1.5391	NaBr·2H ₂ O
3	40.05	10.49	60.65	4.21	1.5496	NaBr·2H ₂ O
4	34.65	15.45	57.84	6.50	1.5663	NaBr·2H ₂ O
5	30.38	20.43	59.93	6.71	1.5843	NaBr·2H ₂ O
6	23.82	26.87	56.46	9.52	1.6049	NaBr·2H ₂ O
7	17.41	32.79	55.09	11.08	1.6242	NaBr·2H ₂ O
8	10.80	38.47	53.04	12.91	1.6488	NaBr·2H ₂ O
9	6.76	43.23	50.73	15.14	1.6723	NaBr·2H ₂ O
10, Q	4.18	47.81	7.78	51.15	1.6846	NaBr·2H ₂ O + MgBr ₂ ·6H ₂ O
11	2.72	48.95	2.01	52.78	1.6795	MgBr ₂ ·6H ₂ O
12	1.32	50.28	0.98	54.15	1.6705	MgBr ₂ ·6H ₂ O
13, N	0.00	51.62	ND	ND	1.6584	MgBr ₂ ·6H ₂ O

^aStandard uncertainties $u(T) = 0.3$ K, $u_r(p) = 0.05$, $u_r(\text{Mg}^{2+}) = 0.0072$, $u_r(\text{Br}^-) = 0.0037$, and $u_r(\rho) = 0.002$. ^bw₁, mass fraction of NaBr; w₂, mass fraction of MgBr₂. ^cND, not determined. N, Q, and H have the same meaning as described in Figure 4.

FIGURE 3: Equilibrium phase diagram of the ternary system NaBr + MgBr₂ + H₂O at 313.15 K. ●, equilibrium liquid phase composition; ■, moist solid phase composition; A, pure solid of NaBr; D, pure solid of MgBr₂; C, pure solid of NaBr·2H₂O; M, pure solid of MgBr₂·6H₂O; W, water; H, solubility of NaBr in water; N, solubility of MgBr₂ in water; Q, cosaturated point of MgBr₂·6H₂O and NaBr·2H₂O.

salting out. In this system, it illustrates that MgBr₂ has a strong salting-out effect on NaBr.

In Figure 3, the same method used in Figure 1 is utilized to analyze the equilibrium solid phase of the system NaBr

+ MgBr₂ + H₂O. Consequently, curve NQ corresponding equilibrium solid phase is MgBr₂·6H₂O and curve HQ corresponding equilibrium solid phase is NaBr·2H₂O. WNQH denotes unsaturated region at 313.15 K. NQM denotes crystallization region of MgBr₂·6H₂O, while HQC denotes crystallization region of NaBr·2H₂O. Zone MQC denotes the mixed crystalline region of MgBr₂·6H₂O + NaBr·2H₂O. It is obvious that crystallization region of MgBr₂·6H₂O is much smaller than that of NaBr·2H₂O.

The phase diagram of the ternary system NaBr + MgBr₂ + H₂O has been studied at 323 K and 348 K [11, 12]. Compared with the three phase diagrams at different temperatures, the result shows that the solubility of MgBr₂·6H₂O is highest at three temperatures. But the numbers of invariant points, crystallization fields, and univariant curves are different. The quaternary systems at 313 K and 348 K are all simple cosaturation type without complex salt and solid solution. They all include one invariant point, two univariant curves, and two crystallization regions (MgBr₂·6H₂O and NaBr·2H₂O at 313 K, MgBr₂·6H₂O and NaBr at 348 K). The phase diagram at 323 K includes two invariant points, three univariant curves, and three crystallization regions, where the solids are NaBr·2H₂O, NaBr, and MgBr₂·6H₂O, respectively.

Figure 4 indicates the relationship between the mass fraction of MgBr₂ and the density in the solution. With an increase of the MgBr₂ concentration, the density first increases and then, the density declines afterwards. At the invariant point Q, the density reaches a maximum value.

4. Conclusions

The phase equilibria in the NaBr + KBr + H₂O and NaBr + MgBr₂ + H₂O ternary systems at 313.15 K were investigated. The solubility and density data of the ternary systems were

FIGURE 4: Density versus $100w$ (MgBr_2) in the ternary system ($\text{NaBr} + \text{MgBr}_2 + \text{H}_2\text{O}$). N, Q, and H have the same meaning as described in Figure 3.

obtained. The diagrams of density versus composition and the ternary phase diagrams were plotted. The equilibrium solid phases were analyzed and the crystalline regions were determined. In ternary system $\text{NaBr} + \text{KBr} + \text{H}_2\text{O}$, the crystalline region of KBr is much larger than that of $\text{NaBr} \cdot 2\text{H}_2\text{O}$ and NaBr has a strong salting-out effect on KBr . In ternary system $\text{NaBr} + \text{MgBr}_2 + \text{H}_2\text{O}$, the crystalline region of $\text{NaBr} \cdot 2\text{H}_2\text{O}$ is much larger than that of $\text{MgBr}_2 \cdot 6\text{H}_2\text{O}$ and MgBr_2 has a strong salting-out effect on NaBr . There are in all two crystalline regions, one invariant point, and two univariant curves in the ternary phase diagrams. All results can offer fundamental data support for optimizing the processes and further theoretical studies.

Conflicts of Interest

The authors declare that there are no financial conflicts of interest.

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