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PHYSICOCHEMICAL ANALYSIS OF INORGANIC SYSTEMS

Liquid-Solid Equilibria in the Quinary System Na^+, K^+||Br^-, SO_4^{2-}, $B_4O_7^{2-}$–H_2O at 298 K[1]

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Abstract—Solid-liquid equilibria in the quinary system Na^+, K^+||Br^-, SO_4^{2-}, $B_4O_7^{2-}$–H_2O at 298 K were measured by isothermal solution saturation method. The solubilities of salts and the densities of saturated solutions in the system were determined. Using the experimental data, phase diagram, Na^+ content diagram, water content diagram and density-composition diagram were constructed, respectively. The double salt $Na_2SO_4 \cdot 3K_2SO_4$ (Gla) was found in the quinary system Na^+, K^+||Br^-, SO_4^{2-}, $B_4O_7^{2-}$–H_2O at 298 K. In the phase diagram, there are eleven univariant curves, five invariant points and seven crystallization regions saturated with $Na_2B_4O_7 \cdot 10H_2O$, where the solids are Na_2SO_4, K_2SO_4, $Na_2SO_4 \cdot 3K_2SO_4$ (Gla), $Na_2SO_4 \cdot 10H_2O$, $NaBr \cdot 2H_2O$, KBr and $K_2B_4O_7 \cdot 4H_2O$. $NaBr \cdot 2H_2O$ has the highest concentration and strong salting-out effects on other salts.

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The underground brines in Western Sichuan Basin (China) are very rare liquid mineral resources in the world. These rare liquid mineral resources have very good exploitation prospect due to the gradual exhaustion of solid resources. The brines contain high concentration of potassium and borate. Its salinity is average up to 377 g L^{-1}, and this brine is weak acidity with pH value of 6.18. In addition to K^+, B^{3+}, the brine still has some useful components such as Br^-, I^-, Li^+, Sr^{2+}, etc, and all of them can reach or exceed the industrial index. The highest content of potassium (K^+) is as high as 53.27 g L^{-1}, much higher than those in the Qarhan salt lake brine in Qinghai, China (12.1 g L^{-1}), the Zabuye salt lake brine in Tibet, China (27.0 g L^{-1}), and the Searls salt lake brine (USA) (23.1 g L^{-1}). The borate concent is up to 4994.36 mg L^{-1} and the bromine content is up to 2533 mg L^{-1} in Sichuan Basin [1–3]. Generally speaking, phase equilibria and phase diagrams are the theoretical basis of the exploitation and utilization of underground brine resources. In view of the abundant borate resources, a series of studies have been conducted on the K^+, Br^- and $B_4O_7^{2-}$-bearing phase equilibria, i.e., $Mg_2B_4O_7$–Mg_2SO_4–H_2O at 298 K [4], KCl–KBr–$K_2B_4O_7$–H_2O at 323 K and 373 K [5, 6], NaCl–NaBr–$Na_2B_4O_7$–H_2O at 348 K [7], and Na–K–Cl–SO_4–H_3BO_3–H_2O at 298 K [8].

Recently, more attention is focused on the solid-liquid equilibrium processes in underground brine system. The phase equilibria of some ternary, quaternary and quinary subsystems of the underground brine in Western Sichuan basin have been carried out in a systematic research program by our group: KBr–$K_2B_4O_7$–H_2O 298 K, 323 K, 348 K, and 373 K [9–12], quaternary system, and $Na_2B_4O_7$–Na_2SO_4–NaBr–H_2O at 298 K, 323 K and 348 K [13–15] and Na–K–Cl–SO_4–B_4O_7–H_2O at 298 K and 323 K [16, 17].

According to the main components in the underground brines in Western Sichuan Basin, it can be approximately described with the quinary system Na^+, K^+||Br^-, SO_4^{2-}, $B_4O_7^{2-}$–H_2O after the salt NaCl is supersaturated and precipitated. Its ternary subsystems $K_2B_4O_7$–KBr–H_2O, $Na_2B_4O_7$–NaBr–H_2O and quaternary subsystems Na^+, K^+||Br^-, $B_4O_7^{2-}$–H_2O, NaBr–Na_2SO_4–$Na_2B_4O_7$–H_2O and KBr–K_2SO_4–$K_2B_4O_7$–H_2O at 298 K have been reported by us [9, 18–20]. In this paper, the quinary system Na^+, K^+||Br^-, SO_4^{2-}, $B_4O_7^{2-}$–H_2O has been researched at room temperature 298 K on the basis of our previous work. So far, there is no research report about the

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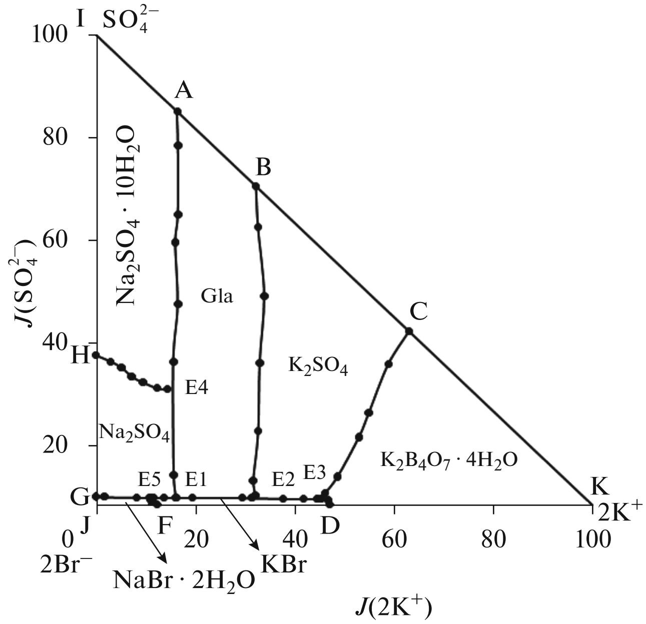

Fig. 1. Dry-salt solubility diagram of the quinary system Na^+, $K^+\|Br^-$, SO_4^{2-}, $B_4O_7^{2-}$–H_2O at 298 K (saturated with $Na_2B_4O_7 \cdot 10H_2O$).

phase equilibria of this system at 298 K, which is just the object of this work.

EXPERIMENTAL

Reagents and Instruments

Distilled water with an electrical conductivity less than 1.2×10^{-4} S m^{-1} and pH 6.6, and the water was used to produce the experimental solutions.

All chemicals used in this work were of analytical grade. There were NaBr, KBr, Na_2SO_4, K_2SO_4, $Na_2B_4O_7 \cdot 10H_2O$, $K_2B_4O_7 \cdot 4H_2O$ (Chengdu Kelong Chemical Reagent Manufactory, China).

An HZS-H type thermostated vibrator with a precision ±0.1 K was used for the equilibrium measurements. A standard analytical balance (AL104) of 110 g capacity, made by the Mettler Toledo Instruments Co., Ltd. with 0.0001 g precision was employed for the determination of the solution density.

Experimental Method

The experiments for the quinary system have been done by the method of isothermal solution saturation. According to the equilibrium composition, salts were prepared proportionally. The appropriate calculated quantity of salts was dissolved in distilled water, but the salt cannot be dissolved thoroughly. The mixtures were put into a sealed glass bottle for the solubility experiments.

Then, sealed glass bottles were placed in the thermostated vibrator (HZS-H), and the temperature was maintained at (298 ± 0.1) K. The solid-liquid systems in sealed tubes were stirred for over a week. The clarification of the solutions needs about five days. The solutions were taken out periodically to analyze the concentration of the solution. When the concentration was unchanged, the sign of equilibrium reached. After equilibrium, the liquid phases were taken out and diluted to volumetric flask for the quantitative analysis of the composition. The densities of the solutions were determined using a pycnometer with an uncertainty of 0.0002 g cm^{-3}.

Analytical Methods

Potassium ion (K^+) concentration was determined using sodium tetraphenyl borate–hexadecyl trimethyl ammonium bromide titration (with a precision of 0.5 wt %). The borate ion ($B_4O_7^{2-}$) was determined by basic titration in the presence of mannitol with phenolphthalein solution as indicator (uncertainty of 0.3 wt %). The (SO_4^{2-}) concentration was measured by a method of alizarin red S volumetry (uncertainty of 0.5 wt %). The bromide ion concentration (Br^-) was determined by Mohr's method with a silver nitrate standard solution (precision: ±0.3%). The sodium ion concentration (Na^+) was evaluated on an ion balance.

RESULTS AND DISCUSSION

The phase equilibria in the quinary system Na^+, $K^+\|Br^-$, SO_4^{2-}, $B_4O_7^{2-}$–H_2O was investigated at 298 K using the isothermal dissolution equilibrium method. The experimental solubilities of salts of saturated solutions are presented in Table 1. The solubility diagram of the quinary system is expressed with Jänecke dry-salt indices, with $J(2K^+) + J(2Br^+) + J(SO_4^{2-}) = 100$ mol. On the basis of Jänecke index values of $J(2K^+)$ and $J(SO_4^{2-})$, the corresponding dry salt phase diagram of the quinary system Na^+, $K^+\|Br^-$, SO_4^{2-}, $B_4O_7^{2-}$–H_2O at 298 K (saturated with $Na_2B_4O_7 \cdot 10H_2O$) is shown in Fig. 1.

The quinary system Na^+, $K^+\|Br^-$, SO_4^{2-}, $B_4O_7^{2-}$–H_2O at 298 K consists of seven crystallization fields, eleven univariant curves, and five invariant points.

The seven crystallization fields correspond to six single salts, Na_2SO_4 (HGE5E1E4 field), K_2SO_4 (BE2E3C field), $Na_2SO_4 \cdot 10H_2O$ (IHE4A field), $NaBr \cdot 2H_2O$ (GJFE5 field), KBr (E5FDE3E2 field), $K_2B_4O_7 \cdot 4H_2O$ (CE3DK field) and one field of double salt, $Na_2SO_4 \cdot 3K_2SO_4$ (AE4E1E2B field).

Solubilities and densities of the quinary system Na^+, $K^+\|Br^-$, SO_4^{2-}, $B_4O_7^{2-}-H_2O$ at 298 K (saturated with $Na_2B_4O_7 \cdot 10H_2O$)

No.	Composition of solution 100 *w* (b)					Janecke index *J*, mol 100 mol^{-1} ($J(2K^+) + J(2Br^+) + J(SO_4^{2-}) = 100$ mol)					Solid phase	Density ρ, g cm^{-3}
	Na^+	K^+	Br^-	SO_4^{2-}	$B_4O_7^{2-}$	K_2^{2+}	Br_2^{2-}	SO_4^{2-}	H_2O	Na_2^{2+}		
1A	7.05	2.77	0.00	17.45	1.10	16.31	0.00	83.69	1831.43	70.65	NB + S10 + Gla	1.3315
2	6.99	2.80	2.42	15.94	1.04	16.51	6.99	76.50	1811.86	70.09	NB + S10 + Gla	1.3931
3	6.91	2.76	7.42	12.73	1.03	16.49	21.66	61.85	1790.17	70.10	NB + S10 + Gla	1.4118
4	6.97	2.66	9.60	11.47	0.97	15.95	28.14	55.91	1775.24	71.02	NB + S10 + Gla	1.4487
5	6.88	2.75	13.94	8.79	0.96	16.46	40.78	42.76	1730.22	69.97	NB + S10 + Gla	1.4659
6	7.76	2.86	20.30	6.92	0.97	15.53	53.88	30.58	1440.71	71.58	NB + S10 + Gla	1.4521
7	7.94	2.96	30.16	1.51	0.94	15.63	77.90	6.47	1293.93	71.26	NB + NS + Gla	1.5218
8E1	7.56	2.93	30.69	0.37	0.94	16.07	82.30	1.63	1367.24	70.46	NB + NS + Gla + KBr	1.5552
9B	3.23	4.14	0.00	10.70	1.82	32.18	0.00	67.82	2705.82	42.76	NB + Gla + KS	1.3163
10	3.18	4.17	2.18	9.29	1.90	32.60	8.32	59.08	2688.69	42.28	NB + Gla + KS	1.3010
11	3.11	4.27	5.73	7.09	1.97	33.25	21.81	44.95	2628.45	41.21	NB + Gla + KS	1.3148
12	3.20	4.25	9.70	4.80	2.06	32.94	36.80	30.25	2555.83	42.17	NB + Gla + KS	1.3527
13	3.43	4.55	14.74	2.68	1.97	32.64	51.71	15.65	2258.87	41.82	NB + Gla + KS	1.3725
14	3.97	5.08	20.74	1.02	1.68	31.63	63.21	5.16	1824.94	42.02	NB + Gla + KS	1.4505
15	4.44	6.00	25.25	0.47	1.62	32.01	65.95	2.04	1441.16	40.34	NB + Gla + KS	1.5399
16E2	4.79	6.18	27.01	0.37	1.61	31.35	67.12	1.53	1323.07	41.40	NB + Gla + KS + KBr	1.5793
17C	0.69	7.48	0.00	5.39	8.46	63.03	0.00	36.97	2853.84	9.86	NB + KS + KB	1.3042
18	0.89	7.16	2.62	4.30	7.71	59.93	10.75	29.32	2807.99	12.62	NB + KS + KB	1.3041
19	0.98	6.89	6.31	2.92	6.13	55.78	24.99	19.23	2695.39	13.44	NB + KS + KB	1.3456
20	1.03	7.08	8.90	2.38	5.02	52.94	32.58	14.47	2454.21	13.05	NB + KS + KB	1.3727
21	1.40	7.99	15.25	1.21	3.81	48.59	45.41	6.00	1856.83	14.50	NB + KS + KB	1.4405
22	1.82	9.21	21.02	0.62	2.99	46.04	51.45	2.51	1396.56	15.45	NB + KS + KB	1.5266
23E3	2.14	10.38	25.78	0.38	2.18	44.53	54.13	1.34	1100.99	15.64	NB + KS + KB + KBr	1.5797
24H	8.77	0.00	20.38	5.71	0.58	0.00	68.20	31.80	1915.99	102.00	NB + NS + S10	1.5033
25	8.36	0.45	20.15	5.55	0.58	3.00	66.53	30.47	1900.43	95.96	NB + NS + S10	1.5227
26	8.30	0.79	20.63	5.52	0.62	5.11	65.68	29.21	1810.91	91.80	NB + NS + S10	1.5453
27	8.03	1.12	20.85	5.23	0.65	7.20	65.50	27.30	1786.03	87.71	NB + NS + S10	1.5697

Table. (Contd.)

No.	Composition of solution 100 *w* (b)					Janecke index *J*, mol 100 mol^{-1} ($J(2K^+) + J(2Br^+) + J(SO_4^{2-}) = 100$ mol)					Solid phase	Density ρ, g cm^{-3}
	Na^+	K^+	Br^-	SO_4^{2-}	$B_4O_7^{2-}$	K_2^{2+}	Br_2^{2-}	SO_4^{2-}	H_2O	Na_2^{2+}		
28	7.67	1.48	20.67	5.03	0.62	9.44	64.45	26.11	1784.60	83.13	NB + NS + S10	1.5911
29	7.14	1.91	20.06	4.78	0.67	12.24	62.84	24.92	1817.40	77.68	NB + NS + S10	1.6576
30E4	6.81	2.26	19.65	4.78	0.68	14.34	60.98	24.67	1811.71	73.49	NB + NS + S10 + Gla	1.6787
31G	11.02	0.00	37.06	0.39	0.57	0.00	98.26	1.74	1198.05	101.56	NB + NS + NBr	1.6702
32	10.73	0.31	36.71	0.38	0.58	1.69	96.66	1.65	1197.69	98.21	NB + NS + NBr	1.6716
33	10.42	1.69	38.39	0.39	0.62	8.12	90.37	1.51	1012.40	85.27	NB + NS + NBr	1.6842
34	9.89	2.28	37.81	0.39	0.57	10.81	87.67	1.52	1008.67	79.72	NB + NS + NBr	1.7254
35	9.28	2.46	36.04	0.39	0.58	12.07	86.37	1.57	1089.09	77.29	NB + NS + KBr	1.7305
36	9.13	3.05	36.67	0.39	0.60	14.30	84.20	1.50	1021.35	72.82	NB + NS + KBr	1.7146
37	8.90	3.61	37.00	0.41	0.62	16.38	82.12	1.50	973.20	68.66	NB + NS + KBr	1.6935
38	8.55	4.65	37.90	0.40	0.62	19.76	78.88	1.37	883.91	61.81	NB + Gla + KBr	1.6759
39	8.15	6.23	39.76	0.37	0.66	23.96	74.88	1.16	748.55	53.35	NB + Gla + KBr	1.5958
40F	9.83	2.66	39.00	0.00	0.58	12.24	87.76	0.00	956.29	76.87	NB + NBr + KBr	1.7240
41	9.88	2.39	38.35	0.17	0.57	11.21	88.13	0.66	991.50	78.92	NB + NBr + KBr	1.7178
42	10.03	2.41	38.83	0.22	0.58	11.18	88.00	0.83	963.01	79.01	NB + NBr + KBr	1.7099
43	10.06	2.38	38.78	0.26	0.60	11.04	87.97	0.99	963.62	79.32	NB + NBr + KBr	1.7221
44	9.88	2.40	38.17	0.31	0.56	11.27	87.56	1.17	990.28	78.79	NB + NBr + KBr	1.7133
45	9.99	2.35	38.35	0.37	0.54	10.98	87.62	1.40	980.69	79.33	NB + NBr + KBr	1.6999
46E5	9.98	2.37	38.25	0.39	0.60	11.05	87.47	1.48	981.88	79.31	NB + NBr + KBr + NS	1.7054
47D	1.44	12.01	27.55	0.00	1.94	47.11	52.89	0.00	971.12	9.61	NB + KBr + KB	1.5544
48	1.44	12.18	27.72	0.27	1.67	46.90	52.26	0.84	948.22	9.44	NB + KBr + KB	1.5340
49	1.82	11.54	27.74	0.37	1.50	45.40	53.41	1.19	973.79	12.17	NB + KBr + KB	1.5490
50	1.98	11.04	27.44	0.38	1.33	44.57	54.20	1.23	1013.21	13.58	NB + KBr + KB	1.5508
51	2.62	9.62	27.14	0.38	0.98	41.46	57.21	1.33	1108.01	19.20	NB + KS + KBr	1.5664
52	2.87	9.14	27.17	0.37	0.83	40.20	58.46	1.34	1137.53	21.43	NB + KS + KBr	1.5775

S10, $Na_2SO_4 \cdot 10H_2O$; Gla, $Na_2SO_4 \cdot 3K_2SO_4$; NS, Na_2SO_4, KS, K_2SO_4; KB, $K_2B_4O_7 \cdot 4H_2O$; NB, $Na_2B_4O_7 \cdot 10H_2O$.

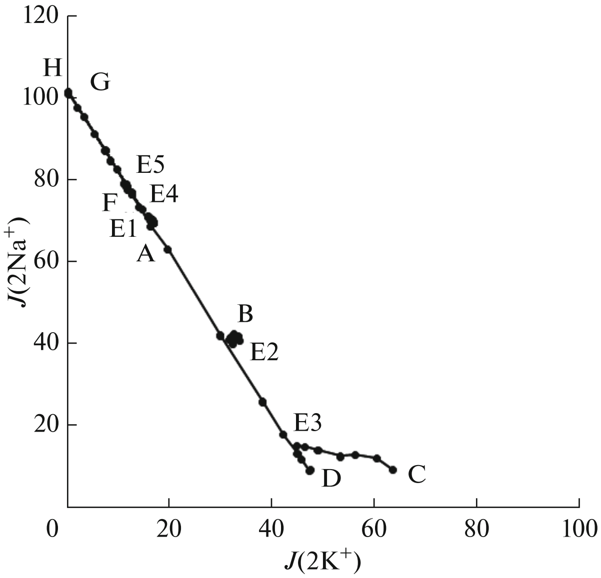

Fig. 2. Na^+ content diagram of the quinary system Na^+, $K^+\|Br^-$, SO_4^{2-}, $B_4O_7^{2-}-H_2O$ at 298 K (saturated with $Na_2B_4O_7 \cdot 10H_2O$).

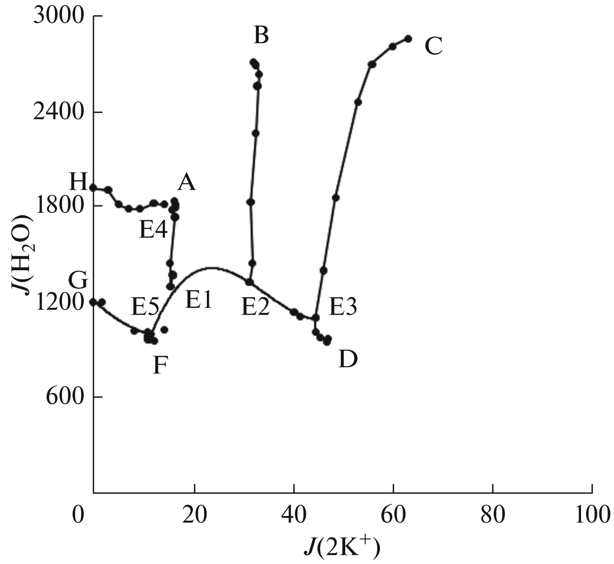

Fig. 3. Water contents of saturated solutions in the quinary system Na^+, $K^+\|Br^-$, SO_4^{2-}, $B_4O_7^{2-}-H_2O$ at 298 K (saturated with $Na_2B_4O_7 \cdot 10H_2O$).

Points A, B, C are three invariant points of the quaternary subsystem Na^+, $K^+\|SO_4^{2-}$, $B_4O_7^{2-}-H_2O$ of this quinary system, points D, F are two invariant points of the quaternary subsystem Na^+, $K^+\|Br^-$, $B_4O_7^{2-}-H_2O$ of this quinary system, and points G, H are the two invariant points of the quaternary subsystem $Na^+\|Br^-$, SO_4^{2-}, $B_4O_7^{2-}-H_2O$ of this quinary system.

The eleven univariant curves are AE4, HE4, E4E5, GE5, FE5, E5E1, E1E2, BE2, E2E3, DE3, and CE3. The five invariant points of this quinary system are labeled as E1, E2, E3, E4 and E5. The saturated salts and the mass fraction composition of the corresponding liquid phase for the invariant points are listed below.

Invairant point E1, saturated with salts Na_2SO_4 + Gla ($Na_2SO_4 \cdot 3K_2SO_4$) + KBr + $Na_2B_4O_7 \cdot 10H_2O$, with $w(Na^+) = 0.0756$, $w(K^+) = 0.0293$, $w(Br^-) = 0.3069$, $w(SO_4^{2-}) = 0.0037$, $w(B_4O_7^{2-}) = 0.0094$.

Invairant point E2, saturated with salts KBr + Gla ($Na_2SO_4 \cdot 3K_2SO_4$) + K_2SO_4 + $Na_2B_4O_7 \cdot 10H_2O$, with $w(Na^+) = 0.0479$, $w(K^+) = 0.0618$, $w(Br^-) = 0.2701$, $w(SO_4^{2-}) = 0.0037$, $w(B_4O_7^{2-}) = 0.0161$.

Invairant point E3, saturated with salts K_2SO_4 + KBr + $K_2B_4O_7 \cdot 4H_2O$ + $Na_2B_4O_7 \cdot 10H_2O$, with $w(Na^+) = 0.0214$, $w(K^+) = 0.1038$, $w(Br^-) = 0.2578$, $w(SO_4^{2-}) = 0.0038$, $w(B_4O_7^{2-}) = 0.0218$.

Invairant point E4, saturated with salts $Na_2SO_4 \cdot 10H_2O$ + Gla ($Na_2SO_4 \cdot 3K_2SO_4$) + Na_2SO_4 + $Na_2B_4O_7 \cdot 10H_2O$, with $w(Na^+) = 0.0681$, $w(K^+) = 0.0226$, $w(Br^-) = 0.1965$, $w(SO_4^{2-}) = 0.0478$, $w(B_4O_7^{2-}) = 0.0068$.

Invairant point E5, saturated with salts $NaBr \cdot 2H_2O$ + KBr + Na_2SO_4 + $Na_2B_4O_7 \cdot 10H_2O$, with $w(Na^+) = 0.0998$, $w(K^+) = 0.0237$, $w(Br^-) = 0.3825$, $w(SO_4^{2-}) = 0.0039$, $w(B_4O_7^{2-}) = 0.0060$.

Figure 2 is the Na^+ contents in this quinary system, and the abscissa is the Jänecke index values of $J(2K^+)$. Figure 2 show that the Na^+ content decreases regularly and researches the minimum value at the point D. The water content diagram of the quinary system is constructed in Fig. 3. Figure 3 shows that the water content decreases apparently from A−E1, B−E2, C−E3, H−E4 and G−E5. Figure 4 is the density diagram of the quinary system. It shows that equilibrium solution density values change with the Jänecke index values of $J(2K^+)$ and reach the largest value (1.7240 g cm^{-3}) at point F.

CONCLUSIONS

The solid–liquid equilibria in the quinary system Na^+, $K^+\|Br^-$, SO_4^{2-}, $B_4O_7^{2-}-H_2O$ at 298 K was studied by the isothermal solution saturation method. Solubilities, densities and corresponding equilibrium solids were determined. The double salt $Na_2SO_4 \cdot 3K_2SO_4$ were found in the quinary system, there are five invari-

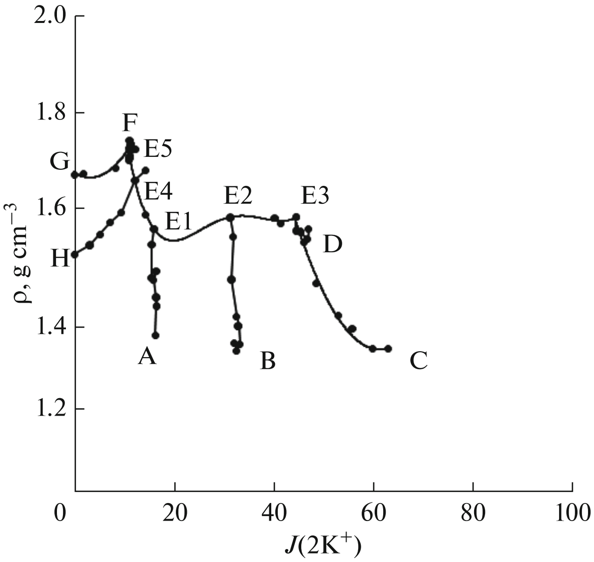

Fig. 4. Density-composition relations of the solutions the quinary system Na^+, K^+||Br^-, SO_4^{2-}, $B_4O_7^{2-}$—H_2O at 298 K (saturated with $Na_2B_4O_7 \cdot 10H_2O$).

ant points, eleven uninvariant curves, and seven crystallization fields (saturated with $Na_2B_4O_7 \cdot 10H_2O$): Na_2SO_4, K_2SO_4, $Na_2SO_4 \cdot 3K_2SO_4$ (Gla), $Na_2SO_4 \cdot 10H_2O$, $NaBr \cdot 2H_2O$, KBr and $K_2B_4O_7 \cdot 4H_2O$.

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