

Solubility and Metastable Zone Width Measurement of Borax Decahydrate in Potassium Chloride Solution

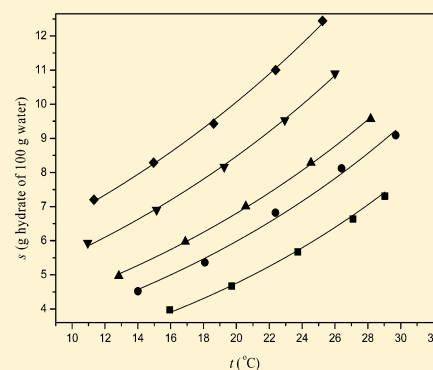
Jiaoyu Peng,^{†,‡} Yaping Dong,^{*,†} Zhen Nie,[§] Fanzhi Kong,^{†,‡} Qingfen Meng,[†] and Wu Li[†]

[†]Qinghai Institute of Salt Lakes, Chinese Academy of Sciences, 810008, Xining, China

[‡]Graduate University of Chinese Academy of Sciences, 100039, Beijing, China

[§]Chinese Academy of Geological Sciences, 100037, Beijing, China

ABSTRACT: The solubility and ultra solubility of borax decahydrate both in pure and impure solutions have been determined in the temperature range from (0 to 30) °C by means of the conventional polythermal method using the laser technique. The metastable zone width and the apparent nucleation order of borax were calculated. The effects and the action mechanism of KCl on metastable zone property of borax were studied. It was found that the presence of KCl made an increase in the solubility and a decrease in the metastable zones of borax. However, the trends in the changes of metastable zone width at low impurity concentrations were different from high concentrations because of the adsorption equilibria on the crystals.



INTRODUCTION

Borax is one of the most important boron compounds. It has been widely used in the glass industry, ceramic industry, nuclear industry, high-energy fuels, and agriculture, and so forth.¹ The boron reserves present in nature are in the forms of solid and liquid. The liquid boron is an important mineral resource and is mainly enriched in brine, seawater, and hot springs.² Increased interest has been given to the exploitation of the liquid boron resource. In the early 1990s, the boron rich in Searles Lake in the United States has been exploited for the production of borax and the Atacama Lake in Chile for the production of boric acid.³ In Qinghai-Tibet Plateau, China, there are thousands of salt lakes which are abundant in the boron element. But until now liquid boron has hardly been used for the industrial production of boron compounds.² To exploit the liquid boron reserve, it is necessary to study the property of the metastable zone of borate in salt lakes.

Zabuye Salt Lake, Tibet, is a worldwide-known carbonate-type salt lake. It contains a great quantity of lithium, boron, and potassium resources. Researchers^{4–7} have studied four seasons of brines from Zabuye Salt Lake by isothermal evaporation experiments and found that the brine of Zabuye Salt Lake had a particular crystallization path during evaporation. During the late evaporation process of brine, the element boron was participated as high-grade borax, which is favorable for the production of refined borax. However, the effects of impurities rich in brine on the metastable zone of borax cannot be neglected during the crystallization of borax. Impurities can affect not only nucleation and crystal growth process, but also crystal morphology. Gürbüz and Özdemir⁸ have investigated the effect of Mg²⁺ and Ca²⁺ ions on the metastable zone width of borax during its crystallization with tincal ore in Turkey.

Their results indicated that the Ca²⁺ had a small effect on the metastable zone width of borax while the Mg²⁺ resulted in a great increase of the metastable zone width. But the effect of K⁺ on the metastable zone width of borax has not been reported. To produce a product with high purity and required quality, it is necessary to study the relationship between impurity K⁺ and metastable zone width of borax. In this study, the laser intensity technology^{9,10} has been used to determine the solubility and metastable zone width of borax decahydrate both in pure and impure solutions as a function of cooling/heating rates.

EXPERIMENT SECTION

Materials and Apparatus. The Na₂B₄O₇·10H₂O supplied from Tianjin Damao Chemical Reagent Factory with purities of >99.5 % was recrystallized from aqueous solution. KCl (reference reagent) was provided from Tianjin Yongda Chemical Reagent Development Center. Water (resistivity, 18.25 MΩ·cm) was deionized from a water purification system (UPT-II-20T, Chengdu Ultrapure Technology Co., Ltd.) before experiments. The experiment setup for measuring supersolubility was shown schematically in Figure 1. The crystallizer was a 100 mL triple jacketed glass vessel with an internal diameter of 35 mm, and its temperature control was accomplished by a programmable thermostatic bath (SF-01-T, Ningbo Haishu Seif Experimental Instrument Factory, China). The accuracy of the thermometer was 0.1 °C. A magnetic stirrer was adopted to ensure adequate mixing at a constant rate of 200 rpm. The helium–neon laser apparatus (GZ-2A, Beijing

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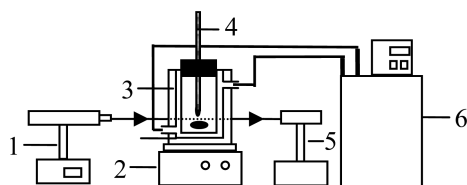


Figure 1. Apparatus for solubility and crystallization measurements: 1, laser generator; 2, electromagnetic stirrer; 3, jacketed glass vessel; 4, precise thermometer; 5, photoelectric converter; 6, programmable thermostatic bath.

TuoDa Laser Instrument Co., Ltd.) was employed to detect nucleation/dissolution by transmitting a light with a wavelength of 632.8 nm into the solution. The X-ray diffraction (XRD) analysis (X'Pert PRO, 2006 PANalytical) was used for confirming the identity of a solid phase crystallizing from the added potassium impurity solutions.

Metastable Zone Width Measurements. The determination of the metastable zone width of the borax in potassium chloride solutions was carried out at the temperature range from (0 to 30) °C according to the conventional polythermal method.¹¹ First, an amount of 80 g of $\text{Na}_2\text{B}_4\text{O}_7\text{-KCl-H}_2\text{O}$ mixture was placed into a 100 mL well-sealed triple jacketed glass vessel. The mixture was then heated and left unsaturated 10 °C above the saturation temperature for 10 min before cooling. Second, the solution was cooled with the settled cooling rate until the first visible nucleus appears. The temperature at the point of nucleation was recorded as t_{nuc} . Last, the mixture was heated at the same constant rate until dissolution of all of the solid phase. The corresponding temperature was recorded as t_{sat} . The above steps were repeated at five cooling/heating rates of (0.20, 0.34, 0.50, 0.64, and 0.80) °C·min⁻¹ and a constant stirring rate of 200 rpm.

During the heating process, however, the closer of the temperature to the point of saturation, the more slowly is the rate of dissolution.^{12,13} For fast heating rates, the temperature measured at the point of disappearance perhaps is greater than the actual saturation temperature. When the heating rate is reduced, the temperature of the point of disappearance approaches the saturation temperature. Therefore, the borax solubility determined by the polythermal method can be obtained by the extrapolation of the measured achievable saturation temperature t_{sat} to a virtual heating rate of "zero".

Table 1 shows a summary of the estimated uncertainties.

Table 1. Uncertainties of Measurements Estimated for This Research

property	estimated uncertainty
solubility	± 0.05 g of 100 g of H ₂ O
saturation temperature	± 0.06 °C
wt % ($\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$)	± 0.08

RESULTS AND DISCUSSION

XRD Analysis. Powder X-ray diffraction (XRD) is useful for confirming the identity of a solid material and phase purity. The XRD analysis was performed using a tube voltage and current of 40 kV and 30 mA, respectively. The scanning position 2θ is from 5.0014° to 69.9754°. Figure 2b shows the XRD spectroscopy of borax decahydrate growing from added

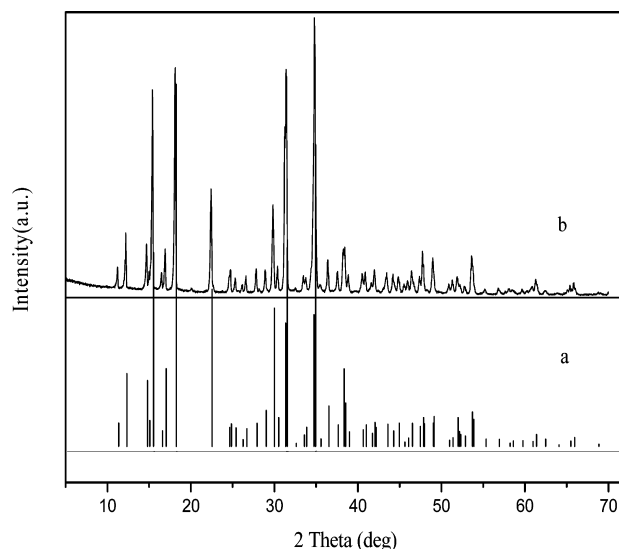


Figure 2. XRD pattern for (a) pure borax decahydrate;¹⁴ (b) potassium.

potassium impurity solutions. Figure 2a shows the pure borax decahydrate pattern obtained from the PDF card (reference code: 01-075-1078), studied by Levy and Lisensky¹⁴ using neutron diffraction data. As seen from the figure, the XRD patterns of pure borax and added potassium chloride borax crystals are identical, demonstrating the K⁺ impurity has not entered into the structure of borax decahydrate crystal. The crystallographic parameters of borax decahydrate are: $C2/c$, $Z = 4$, $a = 11.885(1)$, $b = 10.654(1)$, $c = 12.206(1)$ Å, $\beta = 106.623(5)^\circ$, $\alpha = 90^\circ$, $\gamma = 90^\circ$.¹⁴

Solubility of $\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$ in Potassium Chloride Solutions. The solubility of $\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$ in potassium chloride solutions at various temperatures was determined. The obtained experimental solubility data are shown in Table 2 and

Table 2. Solubility of $\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$ in KCl Solutions

KCl wt %	t °C	solubility of $\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$ in different units	
		wt %	g (of 100 g H ₂ O)
1.84	14.01	4.24	4.52
1.84	18.13	4.99	5.36
1.84	22.38	6.27	6.82
1.84	26.41	7.37	8.12
1.84	29.70	8.18	9.09
3.80	12.84	4.56	4.97
3.80	16.89	5.42	5.97
3.80	20.57	6.30	7.01
3.80	24.54	7.36	8.29
3.80	28.17	8.41	9.57
7.46	10.95	5.18	5.93
7.46	15.14	5.97	6.90
7.46	19.26	6.98	8.16
7.46	22.95	8.05	9.53
7.46	26.00	9.09	10.90
11.19	11.33	5.96	7.20
11.19	14.96	6.80	8.29
11.19	18.61	7.79	9.53
11.19	22.39	8.80	11.00
11.19	25.24	9.82	12.44

demonstrated graphically in Figure 3. It is found that the solubility of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ increases with increasing

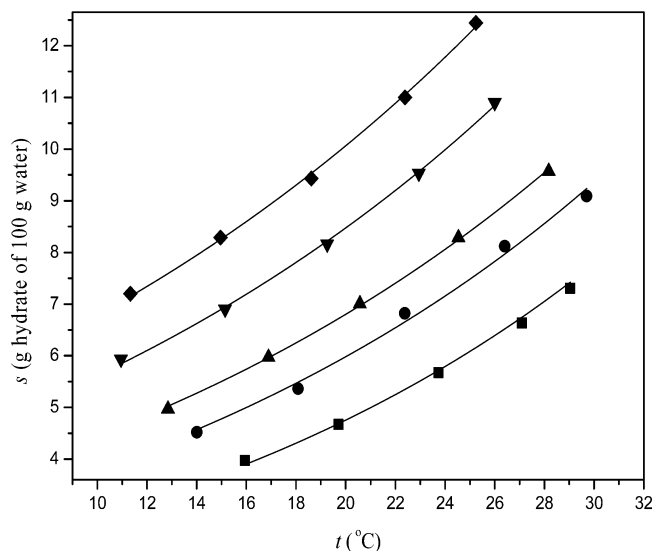


Figure 3. Solubility of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ in KCl solutions determined by the polythermal method. KCl (wt %): ■, 0.00; ●, 1.84; ▲, 3.80; ▼, 7.46; ◆, 11.19.

concentration of potassium chloride. This phenomenon can be mainly attributed to the salt effect of potassium chloride. During the heating stage, there is a dissolution–precipitate equilibrium existing in the dissolution process of borax. The addition of electrolyte potassium chloride can dilute the solute in the solution and leads to a decrease in the effective concentrations of the ions. The equilibrium moves toward the dissolution, thus increasing the solubility of borax.

Metastable Zone Width in Potassium Chloride Solutions. The metastable zone width determination is to obtain a more precise value of the $\Delta t_{\text{max}}/\Delta c_{\text{max}}$ as a crystallizer design choice. However, the metastable zone width is usually influenced by temperature, initial concentration, solution history, cooling rate, stirring, impurities, etc. In this study, the factors of temperature, initial concentration, cooling rate and impurities were considered in the metastable zone width measurement.

From Figure 4 (Table 3) and Figure 5 (Table 4), it is clear that the metastable zone width tends to narrow both with the increase of the temperature and the initial concentration of borax, broadening with an increasing cooling rate. The high temperature can accelerate ion movement and intensify both the ion collision frequency and mass transportation; besides, the concentration of solute increases in high saturation temperature, and the distance between solute molecules becomes shorter, facilitating the earlier appearance of nuclei.

The effect of K^+ impurity on the metastable zone of borax decahydrate was also investigated at the saturation temperature range from (10 to 30) °C. As seen from Figure 6 and Table 5, there is a noticeable decrease of the metastable zone of borax decahydrate with the addition of K^+ impurity. The effect at low K^+ impurity concentrations is much more pronounced than that at high concentrations. Similar results are also found in Figure 7 and Table 6, demonstrating the changes of metastable zone width with K^+ impurity concentrations at saturated solution of 30 °C. It is also shown that the metastable zone width in the impure system gets narrow gradually at low K^+

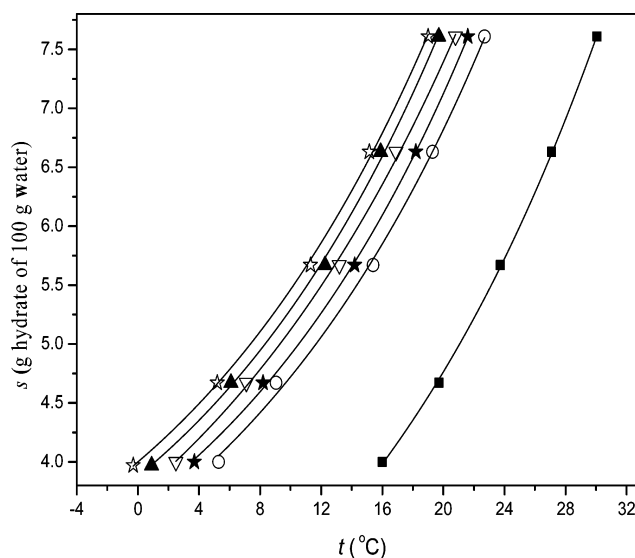


Figure 4. Supersolubility of borax in aqueous solution with different cooling rates. Solubility: ■; Supersolubility: ☆, 0.80 °C·min⁻¹; ▲, 0.64 °C·min⁻¹; ▼, 0.50 °C·min⁻¹; ★, 0.34 °C·min⁻¹; ○, 0.20 °C·min⁻¹.

Table 3. Supersolubility of Borax in Aqueous Solution with Different Cooling Rates^a

c	t_{sat}	t_{nuc}				
		$\beta = 0.80$	$\beta = 0.64$	$\beta = 0.50$	$\beta = 0.34$	$\beta = 0.20$
4.00	16.00	-0.30	0.90	2.50	3.70	6.30
4.67	19.72	5.20	6.10	7.10	8.20	9.05
5.67	23.74	11.30	12.25	13.20	14.20	15.40
6.63	27.10	15.15	15.90	16.90	18.20	19.30
7.61	30.06	19.00	19.70	20.80	20.80	22.70

^a c in g of 100 g water; t_{sat} in °C; t_{nuc} in °C; β in °C·min⁻¹.

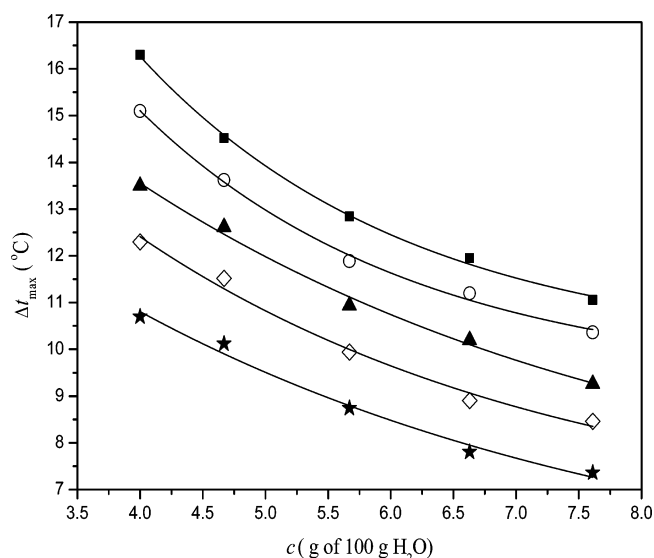


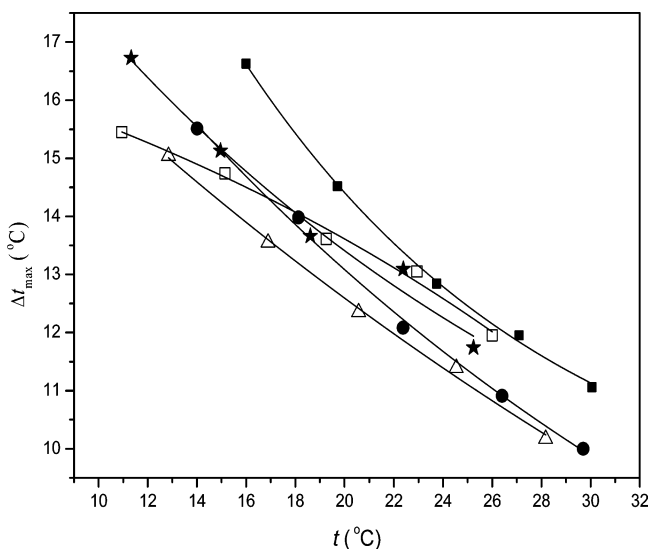
Figure 5. Changes of metastable zone width of borax with initial concentration. ■, 0.80 °C·min⁻¹; ○, 0.64 °C·min⁻¹; ▲, 0.50 °C·min⁻¹; ◇, 0.34 °C·min⁻¹; ★, 0.20 °C·min⁻¹.

impurity concentrations (below 3.80 % KCl). Beyond this concentration the trend is inverted, with the metastable zone width reaching its maximum (about 6.53 % KCl) and falling

Table 4. Changes of the Metastable Zone Width of Borax with Initial Concentration^a

<i>c</i>	Δt_{\max}				
	$\beta = 0.80$	$\beta = 0.64$	$\beta = 0.50$	$\beta = 0.34$	$\beta = 0.20$
4.00	16.30	15.10	13.80	12.30	10.70
4.67	14.52	13.62	12.62	11.52	10.12
5.67	12.84	11.89	10.94	9.94	8.74
6.63	11.95	11.20	10.20	8.90	7.80
7.61	11.06	10.36	9.26	8.46	7.36

^a*c* in g of 100 g water; Δt_{\max} in °C; β in °C·min⁻¹.

**Figure 6. Metastable zone width of borax in KCl solutions at 0.80 °C·min⁻¹. KCl (wt %): ■, 0.00; ●, 1.84; △, 3.80; □, 7.46; ★, 11.19.**

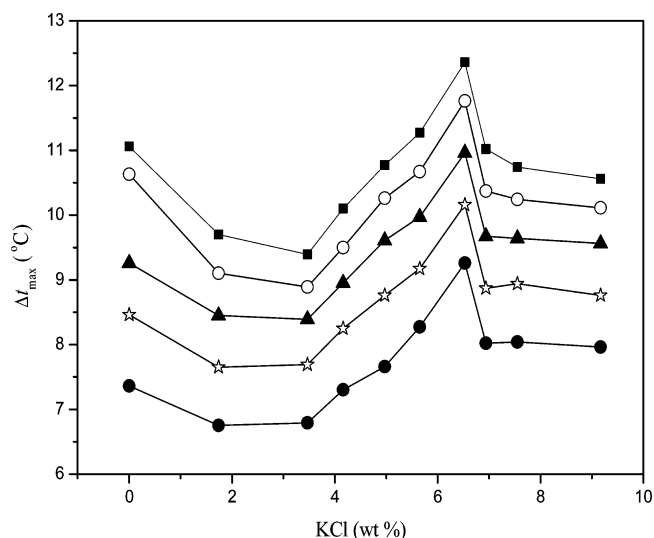
even below that with further impurity addition. All curves tend to level out at higher K⁺ impurity concentrations which means that an even higher K⁺ impurity level would not have a much stronger effect on the metastable zone width.

The impurities affect the width of the metastable zone by different mechanisms.^{15–17} To understand the impact of impurity, one has to distinguish between its effect on nucleation and on growth. Generally, a delay in nucleation and a growth reduction can lead to an increase in metastable zone width. Contrary to this, enhanced nucleation with a moderate growth reduction can reduce the metastable zone width compared to the pure system.¹⁶ Impurities can either enhance the nucleation due to a reduced interfacial tension or suppress it by occupying active growth sites on nuclei.^{18,19} These two opposing effects affect the nucleation together, leading to the nucleation rate

Table 5. Metastable Zone Width of Borax in KCl Solutions at 0.80 °C·min^{-1a}

concentration of KCl (wt %)									
0.00		1.84		3.80		7.46		11.19	
<i>t</i> _{sat}	Δt_{\max}	<i>t</i> _{sat}	Δt_{\max}	<i>t</i> _{sat}	Δt_{\max}	<i>t</i> _{sat}	Δt_{\max}	<i>t</i> _{sat}	Δt_{\max}
16.00	16.63	14.01	15.51	12.84	15.04	10.95	15.45	11.33	16.79
19.72	14.52	18.13	13.98	17.05	13.55	15.14	14.74	14.96	15.13
23.74	12.84	22.38	12.08	20.65	12.35	19.26	13.61	18.61	13.63
27.10	11.95	26.41	10.91	24.67	11.39	22.95	13.07	22.39	13.09
30.06	11.06	29.70	10.00	28.16	10.16	26.00	11.95	25.24	11.74

^a*t*_{sat}, *t*_{nuc} in °C; Δt_{\max} in °C.

**Figure 7. Changes of metastable zone width with impurity concentration at 30 °C. ■, 0.80 °C·min⁻¹; ○, 0.64 °C·min⁻¹; ▲, 0.50 °C·min⁻¹; ☆, 0.34 °C·min⁻¹; ●, 0.20 °C·min⁻¹.****Table 6. Changes of Metastable Zone Width with Impurity Concentration at 30 °C^a**

KCl (wt %)	Δt_{\max}				
	$\beta = 0.80$	$\beta = 0.64$	$\beta = 0.50$	$\beta = 0.34$	$\beta = 0.20$
0.00	11.06	10.36	9.26	8.46	7.36
1.74	9.70	9.10	8.45	7.65	6.75
3.47	9.39	8.89	8.39	7.69	6.79
4.16	10.10	9.50	8.95	8.25	7.30
4.97	10.77	10.26	9.61	8.76	7.66
5.65	11.27	10.67	9.97	9.17	8.27
6.53	12.36	11.76	10.96	10.16	9.26
6.94	11.02	10.37	9.67	8.87	8.02
7.55	10.74	10.24	9.64	8.94	8.04
9.17	10.56	10.11	9.56	8.76	7.96

^a Δt_{\max} in °C; β in °C·min⁻¹.

tent to a point. The influence of impurity on the growth rate can be explained by the adsorption equilibria on crystal surface. In this study, the impact mechanism of K⁺ impurity on metastable zone width is complex. It can both affect the nucleation kinetics and crystal growth rate. As can be clearly seen from Figure 7, the addition of K⁺ impurity can promote the nucleation by reducing the interfacial tension. The metastable zone width tends to narrow because of the enhanced nucleation. With further addition of K⁺ impurities, however, the nucleation rate increases slowly even reduces due

to the occupation of K^+ impurity on the active growth sites on nuclei. The reduction of the growth rate can also be obtained because of the increasing surface coverage of the crystals. Therefore, a suppressed nucleation with a growth reduction leads to an increase in the metastable zone width. On the other hand, it is known that increasing impurities can lead to higher supersaturation.¹⁶ At a high K^+ impurity concentration, the crystal surface is widely covered, and the metastable zone width does not widen as much as the supersaturation rises. The higher supersaturation, in turn, both allows for a faster nucleation rates and growth rates. This may explain why the metastable zone width goes through an extreme point and falling even below that with further more increasing in K^+ impurity concentrations.

Calculation of Apparent Nucleation Order m . According to the classical theory of nucleation,^{11,20,21} the relationship between cooling rates and metastable zone width can be expressed by:

$$\log \Delta t_{\max} = \frac{1-m}{m} \log \left(\frac{dc^*}{dt} \right) - \frac{\log k_n}{m} + \frac{1}{m} \log \beta$$

where β is the cooling rate, k_n is a constant related to the nucleation rate, and m is the apparent nucleation order. Δt_{\max} expresses the metastable zone width. The equation of borax about $\log \Delta t_{\max}$ to $\log \beta$ is summarized in Table 7 and plotted in Figure 8.

From Figure 8 it may be seen that the lines are basically parallel except some deviations because of the measurement errors. It can be corrected by the following formula.¹¹

$$\frac{1}{m} = \frac{\sum_{j=1}^p [\sum_i x_i y_i - \sum_i x_i / N_j \cdot \sum_i y_i]}{\sum_{j=1}^p [\sum_i x_i^2 - (\sum_i x_i)^2 / N_j]}$$

where $x_i = \log \beta_j$, $y_i = \log(\Delta t_{\max})_j$, p is the total number of straight lines, and N_j is the number of measurements carried out for each line. Then the inverse of nucleation order m calculated is 0.2951, and the m of borax is about 3.39 which is in good agreement with the literature value of 3.3.¹¹

Table 7. Nucleation Equation of Borax in Pure Solution

$s_{Na_2B_4O_7 \cdot 10H_2O} / 100 \text{ g water}$	nucleation equation	R^2
4.00	$\log \Delta t_{\max} = 1.2356 + 0.3025 \log \beta$	0.9894
4.67	$\log \Delta t_{\max} = 1.1873 + 0.2775 \log \beta$	0.9986
5.67	$\log \Delta t_{\max} = 1.1291 + 0.2778 \log \beta$	0.9891
6.63	$\log \Delta t_{\max} = 1.1027 + 0.2988 \log \beta$	0.9825
7.61	$\log \Delta t_{\max} = 1.0673 + 0.2947 \log \beta$	0.9840

The apparent nucleation order m of borax under different concentrations of K^+ impurity is illustrated in Table 8. It is noticeable that the nucleation order m of borax is larger than in the pure solution. This suggests that a higher apparent nucleation order of borax can be obtained when K^+ impurity is introduced.

CONCLUSION

The solubility and metastable zone width of borax both in aqueous and potassium chloride solutions have been studied. Because of the salt effect of K^+ impurity, the solubility of borax in pure solution is much lower than impure solution. The higher concentration of K^+ impurity, the greater is the solubility of borax. In all temperatures under consideration, borax has a

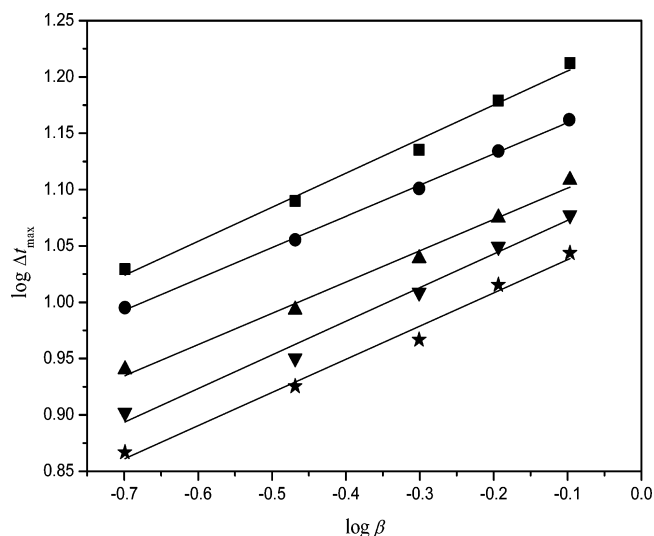


Figure 8. Relationship between $\log \Delta t_{\max}$ and $\log \beta$. c (g/100 g H_2O): ■, 4.00; ●, 4.67; ▲, 5.67; ▼, 6.63.

Table 8. Nucleation Equation of Borax in KCl Solutions

KCl (wt %)	nuclear equation	R^2	m
1.74	$\log \Delta t_{\max} = 1.0135 + 0.2623 \log \beta$	0.9974	3.81
3.47	$\log \Delta t_{\max} = 0.9973 + 0.2329 \log \beta$	0.9992	4.29
4.16	$\log \Delta t_{\max} = 1.0265 + 0.2315 \log \beta$	0.9982	4.32
4.97	$\log \Delta t_{\max} = 1.0991 + 0.2358 \log \beta$	0.9957	4.24
5.65	$\log \Delta t_{\max} = 1.0991 + 0.1963 \log \beta$	0.9901	5.09
6.53	$\log \Delta t_{\max} = 1.1129 + 0.2120 \log \beta$	0.9904	4.72
6.94	$\log \Delta t_{\max} = 1.0547 + 0.2137 \log \beta$	0.9890	4.68
7.55	$\log \Delta t_{\max} = 1.0620 + 0.2283 \log \beta$	0.9887	4.38
9.17	$\log \Delta t_{\max} = 1.0488 + 0.2131 \log \beta$	0.9974	4.69

wide metastable zone width Δt_{\max} . This may be favorable for the choice of the desired crystal size of the product during the industrial crystallization. The effect of K^+ impurity at low concentrations on the metastable zone width of borax is different from high concentrations. The possible mechanism is the adsorption equilibria of the K^+ impurity on the crystal faces. The nucleation order m of borax in aqueous solution is 3.39.

AUTHOR INFORMATION

Corresponding Author

*E-mail: dyp811@126.com. Phone: 86-971-6302023. Fax: 86-971-6310402.

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REFERENCES

- (1) Zheng, X. J. *Production and Application of Boron Compounds*; Chemical Industry Press: Beijing, 2008.
- (2) Gao, S. Y.; Song, P. S.; Xia, S. P.; Zheng, M. P. *Salt Lake Chemistry*; Science Press: Beijing, 2007.
- (3) Song, P. S.; Li, W.; Sun, B.; Nie, Z.; Bu, L. Z.; Wang, Y. S. Recent Development on Comprehensive Utilization of Salt Lake Resources. *Chin. J. Inorg. Chem.* **2011**, *5*, 801–805.
- (4) Nie, Z.; Bu, L. Z.; Zheng, M. P.; Zhang, Y. S. Phase Chemistry Study on Brine from the Zabuye Carbonate-Type Salt Lake in Tibet. *Acta Geol. Sin.* **2010**, *84*, 587–592.

(5) Zheng, M. P.; Deng, Y. J.; Nie, Z.; Bu, L. Z.; Shi, S. H. 25 °C-Isothermal Evaporation of Autumn Brines from Zabuye Salt Lake, Tibet, China. *Acta Geol. Sin.* **2007**, *81*, 1742–1749.

(6) Zhang, Y. S.; Zheng, M. P.; Nie, Z.; Bu, L. Z. 15 °C-Isothermal Evaporation Experiment on Carbonate-type Brine from Zabuye Salt Lake, Tibet, Southwestern China. *J. Salt Chem. Ind.* **2005**, *34*, 1–5.

(7) Yang, J. Y.; Zhang, Y.; Cheng, W. Y.; Jiang, X. C. 25 °C-Isothermal Evaporation of Winter Brines from Zabuye Salt Lake, Tibet. *J. Sal. Chem. Ind.* **1996**, *25*, 21–24.

(8) Gürbüz, H.; Özdemir, B. Experiment Determination of the Metastable Zone Width of Borax Decahydrate by Ultrasonic Velocity Measurement. *J. Cryst. Growth* **2003**, *252*, 343–349.

(9) Meng, Q. F.; Dong, Y. P.; Kong, F. Z.; Feng, H. T.; Li, W. Study on the Metastable Zone Property of Boric Acid in Different Concentrations of MgCl₂ and NaCl Solutions. *Acta Chim. Sin.* **2010**, *68*, 1699–1706.

(10) Chen, Q. L.; Wang, Y. L.; Li, Y. B.; Wang, J. K. Solubility and Metastable Zone of Cefoperazone Sodium in Acetone + Water System. *J. Chem. Eng. Data* **2009**, *54*, 1123–112.

(11) Nývlt, J.; Söhnel, O.; Matuchová, M.; Broul, M. *The Kinetics of Industrial Crystallization*; Elsevier: Amsterdam, 1985.

(12) Patrícia, C.; Carlos, L.; Manuel, E. M.; Piedade, J. A.; Lourenco, A.; Serrano, M. L. Solubility and Metastable Zone Width of 1-Keto-1,2,3,4-tetrahydro-6-methylcarbazole in Acetone. *J. Chem. Eng. Data* **2006**, *51*, 1306–1309.

(13) Barrett, P.; Glennon, B. Characterizing the Metastable Zone Width and Solubility Curve Using Lasentec FBRM and PVM. *Trans. IChemE, Part A* **2002**, *80*, 799–805.

(14) Levy, H. A.; Lisensky, G. C. Crystal Structures of Sodium Sulfate Decahydrate (Glauber's Salt) and Sodium Tetraborate Decahydrate (Borax): Redetermination by Neutron Diffraction. *Acta Crystallogr.* **1978**, *B34*, 3502–3510.

(15) Sangwal, K.; Mielniczek-Brzóska, E. Effect of Impurities on Metastable Zone Width for the Growth of Ammonium Oxalate Monohydrate Crystals from Aqueous Solutions. *J. Cryst. Growth* **2004**, *267*, 662–675.

(16) Rauls, M.; Bartosch, K.; Kind, M.; Kuch, St.; Lacmann, R.; Mersmann, A. The Influence of Impurities on Crystallization Kinetics—a Case Study on Ammonium Sulfate. *J. Cryst. Growth* **2000**, *213*, 116–128.

(17) Sayan, P.; Ulrich, J. Effect of Various Impurities on the Metastable Zone Width of Boric Acid. *Cryst. Res. Technol.* **2001**, *4–5*, 411–417.

(18) Buchfink, R.; Schmidt, C.; Ulrich, J. Fe³⁺ As an Example of the Effect of Trivalent Additives on the Crystallization of Inorganic Compounds, Here Ammonium Sulfate. *CrystEngComm* **2011**, *13*, 1118–1122.

(19) Ginde, R. M.; Myerson, A. S. Effect of Impurities on Cluster Growth and Nucleation. *J. Cryst. Growth* **1993**, *126*, 216–222.

(20) Nývlt, J. Kinetics of Nucleation in Solutions. *J. Cryst. Growth* **1968**, *3–4*, 377–383.

(21) Mullin, J. W. *Crystallization*, 2nd ed.; Butterworths: London, 1972.