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# Solubility and Phase Diagram for the Quaternary System $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$ at 293 K

**Hong-kun Zhao,* Cao Tang, Dao-sen Zhang, Wen-lin Xu, Ya-qiong Wang, and Pan-ming Jian**

College of Chemistry & Chemical Engineering, Yangzhou University, Yagngzhou, Jiangsu, 225002, People's Republic of China

Solubility data for the $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$ system at 293 K were determined by Schreinmaker's method. The results were used to construct a phase diagram in the oblique projection according to the Jãneck method. At constant temperature, when the hydrogen peroxide concentration is below 50 % (mass fraction), this diagram includes three invariant points, six double saturated liquid curves, and four crystallization fields corresponding to $Na_2CO_3 \cdot 10H_2O$, $Na_2SO_4 \cdot 10H_2O$, $Na_2CO_3 \cdot 1.5H_2O_2 \cdot H_2O$, and $Na_2SO_4 \cdot 0.5H_2O_2 \cdot H_2O$.

## Introduction

Sodium percarbonate is commonly manufactured by the reaction of sodium carbonate with hydrogen peroxide and is a good candidate to replace sodium perborate tetrahydrate and chlorine bleaching agents, because it has the advantage of greater solubility, greater active oxygen content, and does not damage the fabric, and so forth.[1−8] Interest in the use of sodium percarbonate to replace sodium perborate tetrahydrate and chlorine bleaching agents in detergents is increasing. Sodium sulfate is usually added into the mother solution as a salting-out agent.[9−11] This study on the quaternary system $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$ has given a basis for sodium percarbonate preparation and was necessary for an assessment of the optimum operating parameters for sodium percarbonate crystallization. There are abundant natrum resources in China which can be used for preparing sodium percarbonate. However, natrum not only contains sodium carbonate and sodium bicarbonate but also contains other soluble salts such as sodium sulfate. As such, the cost of using natrum as a raw material will decrease considerably.

The solid−liquid phase equilibrium investigations were based on the determination of the composition of the solution in equilibrium with the solid phase and in salt cocrystallization regions. Those solubility isotherms are commonly presented in a graphical form as the projection of a plane according to the Jãneck method. The diagram allows for the determination of the location and size of the salts cocrystallization regions and of isothermal invariant points that exist in the system.

Experimental results for the system $Na_2CO_3 + H_2O_2 + H_2O$,[12−14] $Na_2SO_4 + H_2O_2 + H_2O$,[12] and $Na_2CO_3 + Na_2SO_4 + H_2O$[12] have been previously published. No research has been reported on the phase diagram of the quaternary system $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$. The objective of this research is to determine the solubility and construct a phase diagram for the quaternary system $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$.

## Experimental Section

***Materials and Apparatus.*** The mass fraction purities of sodium carbonate and sodium sulfate are no less than 99.6 %. The hydrogen peroxide (not containing stabilizers) was purchased from the Jiangsu Yangnong Chemical Group Co. Ltd., with a mass fraction of 50 %. A thermostat water bath was used for controlling measurements at (293 ± 0.1) K. A Rigaku D/max-2400 X-ray diffraction analyzer was used for solid-phase X-ray analysis.

***Procedure.*** Schreinmaker's wet residue method was used in this experiment. The system to be studied was prepared by mixing amounts of the four components to give convenient quantities of solution and of solid phase after establishing the equilibrium condition. The components were taken in such proportions that the composition of the resulting saturated solution fell in the desired portion of the solubility curve. All mixtures were stirred in flasks in a thermostat bath, in which the temperature was controlled at (293 ± 0.1) K. Fifteen identical samples were prepared and placed into a thermostat, and chemical titrations were used to analyze the liquid phase of the samples at 2 h intervals. If the result became constant, then it indicated that equilibrium was achieved. Results indicated that it took about 6 h to reach equilibrium. After equilibrium was accomplished, the solid and the liquid were separated by filtration.

The equilibrium liquid phase and wet residue were dissolved in water and analyzed by titration. The sodium carbonate concentration was determined by titration using standard hydrochloric acid. The hydrogen peroxide concentration was determined by titration using an acidified solution with standard potassium permanganate. The concentration of sodium sulfate was determined by titration using an acidified solution ($pH = 6$, avoiding the affection of the ion $CO_3^{2-}$) with a standard solution of barium chloride in the presence of sodium alizarinsulfonate as an indicator. Each analysis was repeated three times, and the average value of the three measurements was considered as the final value of the analysis; the average relative error of the measurement was estimated to be 0.1 %. In these studies, X-ray diffraction was also employed to determine whether the solid phase was pure and the nature of the compound.

## Results and Discussion

The experimental data for the quaternary system $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$ at 293 K are presented in Table 1.

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* To whom correspondence should be addressed. E-mail: hkzhao@zzu.edu.cn. Tel: +(86) 514 7890481. Fax: +(86) 514 7975244.

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**Table 1. Mass Fraction Solubilities of the Quaternary System Hydrogen Peroxide (1) + Sodium Sulfate (2) + Sodium Carbonate (3) + Water (4) at 293 K**[a]

| mass fraction | | | | Jãneck index/[kg/100 kg of dry salt] | | | | |
|---|---|---|---|---|---|---|---|---|
| liquid phase | | | | liquid phase | | | | |
| 100 $w_1$ | 100 $w_2$ | 100 $w_3$ | 100 $w_4$ | $S_1$ | $S_2$ | $S_3$ | $S_4$ | solid phase |
| | | | | $E_1P_1$ | | | | |
| 0.94 | 1.52 | 24.26 | 73.27 | 3.52 | 5.70 | 90.78 | 274.18 | $N_{10} + A$ |
| 1.19 | 3.16 | 22.41 | 73.24 | 4.45 | 11.81 | 83.74 | 273.69 | $N_{10} + A$ |
| 1.04 | 4.71 | 21.15 | 73.10 | 3.86 | 17.51 | 78.63 | 271.80 | $N_{10} + A$ |
| 1.00 | 5.58 | 20.43 | 72.99 | 3.70 | 20.66 | 75.64 | 270.21 | $N_{10} + A$ |
| 0.80 | 7.20 | 19.07 | 72.93 | 2.94 | 26.61 | 70.45 | 269.45 | $N_{10} + A$ |
| 0.66 | 8.25 | 18.34 | 72.75 | 2.44 | 30.28 | 67.28 | 266.94 | $N_{10} + A$ |
| | | | | $E_2P_1$ | | | | |
| 0.41 | 9.86 | 17.02 | 72.72 | 1.49 | 36.13 | 62.38 | 266.61 | $N_{10} + S_{10}$ |
| | | | | $P_1P_2$ | | | | |
| 0.99 | 9.02 | 17.66 | 72.33 | 3.59 | 32.60 | 261.40 | 261.40 | $S_{10} + A$ |
| 1.16 | 10.24 | 17.42 | 71.17 | 4.04 | 35.53 | 246.89 | 246.89 | $S_{10} + A$ |
| 1.20 | 11.51 | 16.70 | 70.60 | 4.07 | 39.15 | 240.10 | 240.10 | $S_{10} + A$ |
| 1.31 | 14.65 | 14.23 | 69.81 | 4.34 | 48.53 | 231.21 | 231.21 | $S_{10} + A$ |
| 1.09 | 17.45 | 11.76 | 69.70 | 3.59 | 57.58 | 230.06 | 230.06 | $S_{10} + A$ |
| 1.11 | 20.39 | 7.45 | 71.06 | 3.82 | 70.45 | 245.52 | 245.52 | $S_{10} + A$ |
| 2.18 | 20.65 | 5.54 | 71.62 | 7.69 | 72.78 | 252.42 | 252.42 | $S_{10} + A$ |
| 3.12 | 20.40 | 4.00 | 72.48 | 11.34 | 74.13 | 263.37 | 263.37 | $S_{10} + A$ |
| 3.81 | 20.21 | 2.74 | 73.24 | 14.23 | 75.52 | 273.65 | 273.65 | $S_{10} + A$ |
| | | | | $P_2P_3$ | | | | |
| 5.09 | 20.07 | 1.69 | 73.15 | 18.95 | 74.75 | 6.30 | 272.44 | $A + C$ |
| 7.53 | 18.34 | 1.26 | 72.88 | 27.75 | 67.62 | 4.63 | 268.71 | $A + C$ |
| 8.50 | 17.60 | 1.19 | 72.72 | 31.15 | 64.50 | 4.35 | 266.58 | $A + C$ |
| 12.44 | 13.76 | 1.79 | 72.01 | 44.45 | 49.15 | 6.40 | 257.21 | $A + C$ |
| 13.88 | 12.32 | 2.27 | 71.53 | 48.74 | 43.28 | 7.98 | 251.20 | $A + C$ |
| 16.61 | 9.69 | 3.24 | 70.46 | 56.24 | 32.8 | 10.96 | 238.52 | $A + C$ |
| | | | | $E_3P_2$ | | | | |
| 5.74 | 23.05 | 1.29 | 69.92 | 19.09 | 76.63 | 4.28 | 232.41 | $S_{10} + C$ |
| 8.20 | 25.43 | 0.61 | 65.77 | 23.94 | 74.28 | 1.78 | 192.10 | $S_{10} + C$ |
| 4.69 | 20.95 | 1.79 | 72.58 | 17.09 | 76.39 | 6.52 | 264.71 | $S_{10} + C$ |
| | | | | $E_4P_3$ | | | | |
| 21.93 | 6.19 | 4.87 | 67.02 | 66.48 | 18.76 | 14.76 | 203.21 | $A + B$ |
| 20.14 | 7.05 | 4.19 | 68.61 | 64.18 | 22.47 | 13.36 | 218.62 | $A + B$ |
| 18.71 | 8.26 | 3.68 | 69.34 | 61.03 | 26.96 | 12.01 | 226.21 | $A + B$ |
| | | | | $P_3T$ | | | | |
| 19.70 | 10.49 | 3.35 | 66.45 | 58.74 | 31.26 | 10.00 | 198.10 | $B + C$ |
| | | | | $P_1$ | | | | |
| 0.01 | 0.12 | 0.25 | 99.62 | 2.31 | 32.04 | 65.65 | 264.71 | $N_{10} + S_{10} + A$ |
| | | | | $P_2$ | | | | |
| 0.06 | 0.28 | 0.03 | 99.63 | 16.19 | 75.81 | 8.00 | 273.30 | $S_{10} + A + C$ |
| | | | | $P_3$ | | | | |
| 0.26 | 0.13 | 0.05 | 99.56 | 59.41 | 29.31 | 11.28 | 226.21 | $A + C + B$ |

[a] $N_{10}$, $Na_2CO_3 \cdot 10H_2O$; $A$, $Na_2CO_3 \cdot 1.5H_2O_2 \cdot H_2O$; $S_{10}$, $Na_2SO_4 \cdot 10H_2O$; $B$, $Na_2CO_3 \cdot 2H_2O_2 \cdot H_2O$; $C$, $Na_2SO_4 \cdot 0.5H_2O_2 \cdot H_2O$. $w$: Mass fraction. $S$: Jãneck index/[kg/100 kg of dry salt].

The Jãneck index was calculated from

$$S_1 = 100\, w_1/(w_1 + w_2 + w_3)$$

$$S_2 = 100\, w_2/(w_1 + w_2 + w_3)$$

$$S_3 = 100\, w_3/(w_1 + w_2 + w_3)$$

$$S_4 = 100\, w_4/(w_1 + w_2 + w_3)$$

In these equations, $w_1$, $w_2$, $w_3$, and $w_4$ represented the mass concentration of hydrogen peroxide, sodium sulfate, sodium carbonate, and water. $S_1$, $S_2$, $S_3$, and $S_4$ represent the Jãneck index/[kg/100 kg of dry salt] of hydrogen peroxide, sodium sulfate, sodium carbonate, and water, respectively. The data presented in Table 1 and the data representing the Jãneck index were used for construction of the solubility diagram of the studied system.

The phase diagram of the quaternary system $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$ at 293 K is shown in Figures 1 and 2. These curves are defined by a solution saturated with two salts: curve $E_1P_1$, corresponds to the coexistence of the salts $Na_2CO_3 \cdot 10H_2O$ and $Na_2CO_3 \cdot 1.5H_2O_2 \cdot H_2O$; curve $E_2P_1$ corresponds to the coexistence of the salts $Na_2CO_3 \cdot 10H_2O$ and $Na_2SO_4 \cdot 10H_2O$; curve $P_1P_2$ corresponds to the coexistence of the salts $Na_2SO_4 \cdot 10H_2O$ and $Na_2CO_3 \cdot 1.5H_2O_2 \cdot H_2O$; curve $P_2P_3$ corresponds to the coexistence of the salts $Na_2SO_4 \cdot 0.5H_2O_2 \cdot H_2O$ and $Na_2CO_3 \cdot 1.5H_2O_2 \cdot H_2O$; curve $E_3P_2$ corresponds to the coexistence of the salts $Na_2SO_4 \cdot 10H_2O$ and $Na_2SO_4 \cdot 0.5H_2O_2 \cdot H_2O$; and curve $E_4P_3$ corresponds to the coexistence of the salts $Na_2CO_3 \cdot 1.5H_2O_2 \cdot H_2O$ and $Na_2CO_3 \cdot 2H_2O_2 \cdot H_2O$.

In Figure 1, the sides HH′A′A, A′AN′N, and N′NHH′ correspond to the ternary $Na_2SO_4 + H_2O_2 + H_2O$, $Na_2SO_4 + Na_2CO_3 + H_2O$ and $Na_2CO_3 + H_2O_2 + H_2O$ systems at 293 K, respectively (solubilities of the three ternary systems are given in Table 2). Curves $E_3S_A$ and $E_3a$ correspond to the crystallization line of the salts $Na_2SO_4 \cdot 10H_2O$ and $Na_2SO_4 \cdot 0.5H_2O_2 \cdot H_2O$ in the ternary $Na_2SO_4 + H_2O_2 + H_2O$ system,

**Table 2. Mass Fraction Solubilities of the Ternary Systems $Na_2CO_3 + H_2O_2 + H_2O$, $Na_2SO_4 + H_2O_2 + H_2O$, and $Na_2SO_4 + Na_2CO_3 + H_2O$ at 293 K[12]**

| mass fraction of liquid phase | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| $Na_2CO_3$ (1) + $H_2O_2$ (2) + $H_2O$ (3) | | | $Na_2SO_4$ (1) + $H_2O_2$ (2) + $H_2O$ (3) | | | $Na_2SO_4$ (1) + $Na_2CO_3$ (2) + $H_2O$ (3) | | |
| 100 $w_1$ | 100 $w_2$ | 100 $w_3$ | 100 $w_1$ | 100 $w_2$ | 100 $w_3$ | 100 $w_1$ | 100 $w_2$ | 100 $w_3$ |
| 18.00 | 0.00 | 82.00 | 24.62 | 60.80 | 14.58 | 16.32 | 0.00 | 83.68 |
| 19.36 | 0.64 | 80.00 | 25.01 | 59.90 | 15.09 | 12.88 | 5.62 | 81.50 |
| 25.90 | 1.02 | 73.08 | 26.36 | 56.00 | 17.64 | 11.04 | 14.89 | 74.07 |
| 11.93 | 2.31 | 85.76 | 26.63 | 53.60 | 19.77 | 9.41 | 15.29 | 75.30 |
| 5.14 | 9.30 | 85.56 | 26.64 | 52.60 | 20.76 | 0.00 | 17.70 | 82.30 |
| 6.20 | 15.11 | 78.69 | 25.95 | 52.40 | 21.65 | | | |
| 6.04 | 23.83 | 70.13 | 25.19 | 49.08 | 25.73 | | | |
| 7.43 | 29.94 | 62.63 | 24.23 | 44.99 | 30.78 | | | |
| 7.80 | 39.27 | 52.93 | 23.93 | 40.10 | 35.97 | | | |
| 8.07 | 43.06 | 48.87 | 23.89 | 35.51 | 40.6 | | | |
| 7.76 | 58.00 | 34.24 | 24.40 | 30.09 | 45.51 | | | |
| | | | 25.13 | 25.45 | 49.42 | | | |
| | | | 26.17 | 20.88 | 52.95 | | | |
| | | | 28.51 | 13.38 | 58.11 | | | |
| | | | 28.89 | 12.07 | 59.04 | | | |
| | | | 25.95 | 10.78 | 63.27 | | | |
| | | | 21.61 | 7.32 | 71.07 | | | |
| | | | 19.21 | 4.82 | 75.97 | | | |
| | | | 15.75 | 0 | 84.25 | | | |

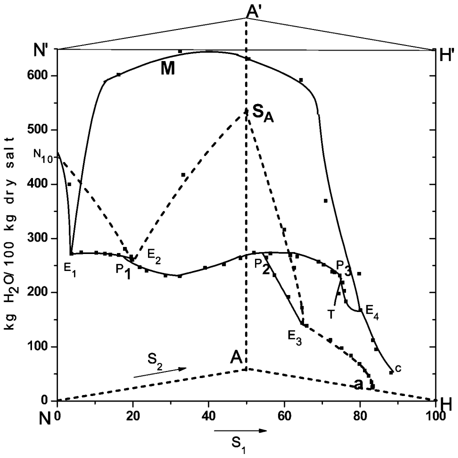

**Figure 1.** Phase diagram of the $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$ system at 293 K. Cosaturated point $E_1$, $Na_2CO_3{\cdot}10H_2O$ and $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$; cosaturated point $E_2$, $Na_2CO_3{\cdot}10H_2O$ and $Na_2SO_4{\cdot}10H_2O$; cosaturated point $E_3$, $Na_2SO_4{\cdot}0.5H_2O_2{\cdot}H_2O$, $Na_2SO_4{\cdot}10H_2O$; cosaturated point $E_4$, $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$ and $Na_2CO_3{\cdot}2H_2O_2{\cdot}H_2O$. Cosaturated point $P_1$, $Na_2SO_4{\cdot}10H_2O$, $Na_2CO_3{\cdot}10H_2O$, and $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$; cosaturated point $P_2$, $Na_2SO_4{\cdot}10H_2O$, $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, and $Na_2SO_4{\cdot}0.5H_2O_2{\cdot}H_2O$; cosaturated point $P_3$, $Na_2CO_3{\cdot}2H_2O_2{\cdot}H_2O$, $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, and $Na_2SO_4{\cdot}0.5H_2O_2{\cdot}H_2O$; The fields I, II, III, and IV correspond to the crystallization zone of the salts $Na_2CO_3{\cdot}10H_2O$, $Na_2SO_4{\cdot}10H_2O$, $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, and $Na_2SO_4{\cdot}0.5H_2O_2{\cdot}H_2O$, respectively, with the saturated solution.

$E_3$ is a cosaturation point with the two solid phases. Similarly, in the ternary $Na_2CO_3 + H_2O_2 + H_2O$ system, curves $N_{10}E_1$, $E_1ME_4$, and $E_4C$ correspond to the crystallization line of the salts $Na_2CO_3{\cdot}10H_2O$, $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, and $Na_2CO_3{\cdot}2H_2O_2{\cdot}H_2O$; $E_1$, $E_4$ are cosaturation points with $Na_2CO_3{\cdot}10H_2O$, $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, and $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, $Na_2CO_3{\cdot}2H_2O_2{\cdot}H_2O$. The curves $N_{10}F_2$ and $F_2S_A$ correspond to the crystallization line of $Na_2CO_3{\cdot}10H_2O$ and $Na_2SO_4{\cdot}10H_2O$, respectively, in the ternary $Na_2SO_4 + Na_2CO_3 + H_2O$ system.

Figure 2 is divided into five crystallization fields. The fields I, II, III, and IV correspond to the crystallization zone of the salts $Na_2CO_3{\cdot}10H_2O$, $Na_2SO_4{\cdot}10H_2O$, $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, and $Na_2SO_4{\cdot}0.5H_2O_2{\cdot}H_2O$, respectively. When the concentration of hydrogen peroxide increases, sodium sulfate will react with hydrogen peroxide to produce the adduct, $Na_2SO_4{\cdot}H_2O_2$; the crystallization field V should include the adducts $Na_2SO_4{\cdot}H_2O_2$ and $Na_2CO_3{\cdot}2H_2O_2{\cdot}H_2O$. In addition, the objective of this investigation was mainly to determine the crystallization region of $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$ with sodium sulfate as a salt-out agent and provide a methodology for preparation of $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$ using natrum as the raw material.

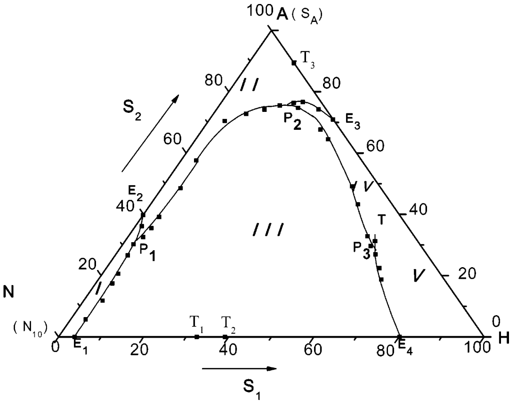

**Figure 2.** Jãneck projection of the quaternary $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$ system at 293 K. $T_1$, $T_2$, and $T_3$, the composition point of the salts $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, $Na_2CO_3{\cdot}2H_2O_2{\cdot}H_2O$, and $Na_2SO_4{\cdot}0.5H_2O_2{\cdot}H_2O$; $S_A$, the solubility of $Na_2SO_4$ in water at 293 K; $N_{10}$, $Na_2CO_3{\cdot}10H_2O$; $E_1$, $E_2$, $E_3$, $E_4$, $P_1$, $P_2$, $P_3$, I, II, III, and IV have the same meaning as those described in Figure 1.

Points $P_1$, $P_2$, and $P_3$ are three isothermal points. Point $P_1$ stands for the solution composition being in equilibrium with the salts $Na_2CO_3{\cdot}10H_2O$, $Na_2SO_4{\cdot}10H_2O$, and $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$; point $P_2$ stands for the solution composition being in equilibrium with the salts $Na_2SO_4{\cdot}10H_2O$, $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, and $Na_2SO_4{\cdot}0.5H_2O_2{\cdot}H_2O$; point $P_3$ stands for the solution composition being in equilibrium with the salts $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$, $Na_2SO_4{\cdot}0.5H_2O_2{\cdot}H_2O$, and $Na_2CO_3{\cdot}2H_2O_2{\cdot}H_2O$.

In Figure 1, $N_{10}E_1P_1E_2$ corresponds to the crystallization area of the salt $Na_2CO_3{\cdot}10H_2O$; $E_1ME_4P_1$ corresponds to the crystallization area of the salt $Na_2CO_3{\cdot}1.5H_2O_2{\cdot}H_2O$; $S_AE_2P_1P_2E_3$ corresponds to the crystallization areas of the salt $Na_2SO_4{\cdot}10H_2O$; $E_3P_2P_3T$ corresponds to the crystallization area of the salt $Na_2SO_4{\cdot}0.5H_2O_2{\cdot}H_2O$.

## Conclusion

In this investigation, solubility data of the $Na_2CO_3 + Na_2SO_4 + H_2O_2 + H_2O$ system at 293 K were determined by

Schreinmaker's method with the concentration of hydrogen peroxide below 50 % (mass fraction). The phase diagram of the quaternary system was constructed according to the Jãneck method. At constant temperature, this phase diagram includes three invariant points, six double saturated liquid curves, and four crystallization fields corresponding to $Na_2CO_3 \cdot 10H_2O$, $Na_2SO_4 \cdot 10H_2O$, $Na_2CO_3 \cdot 1.5H_2O_2 \cdot H_2O$, and $Na_2SO_4 \cdot 0.5H_2O_2 \cdot H_2O$. The crystallization region of $Na_2CO_3 \cdot 1.5H_2O_2 \cdot H_2O$ is greater than that of the other crystals when $Na_2SO_4$ exits in the system $Na_2CO_3 + H_2O_2 + H_2O$, meanwhile the crystallization region of $Na_2CO_3 \cdot 1.5H_2O_2 \cdot H_2O$ declines with the amount of $Na_2SO_4$. The solubility data and the phase diagram of the quaternary system can provide a basis for the preparation of sodium percarbonate.

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