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Solid−Liquid Equilibria of the Ternary Sodium 3-Nitrobenzenesulfonate + Sodium 4-Nitrobenzenesulfonate + Water System

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ABSTRACT: In this investigation, the mutual solubility for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system was determined at (283.15, 303.15, and 323.15) K. The phase diagrams of the system were constructed based on the measured solubility. The solid phases formed in the studied system were confirmed by Schreinemaker's wet residue method. In addition, the density of the equilibrium liquid phase was obtained. At (283.15, 303.15, and 323.15) K, there are two pure solids formed which correspond to sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate in the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system. Besides, the adduct of sodium 3-nitrobenzenesulfonate with sodium 4-nitrobenzenesulfonate, in which the mole ratio of the two compositions is 1:1, was formed at 323.15 K.

INTRODUCTION

Sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate are important intermediate materials. Sodium 3-nitrobenzenesulfonate is widely used for the synthesis of advanced pigments, dyes, ionic solute, and polymers.[1−4] Sodium 4-nitrobenzenesulfonate is widely used in nucleophilic substitution reactions,[5] controlling of the mode of polymorphic transition,[6] and effect of ionic liquids on a class of charge-neutral nucleophiles.[7] They are in general produced by sulfonation of nitrobenzene with concentrated sulfuric acid. The isomeric mixtures are formed in various proportions of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate with this method. The entire usage requires purified sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate products. The commercial product of sodium 3-nitrobenzenesulfonate is obtained via crystallization from the isomeric mixtures of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate in water at present. During the separation process, the mutual solubility of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate in water is needed. The optimization of process conditions is closely related to the solubility of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate in water. It is important to study the system and construct the phase diagram of the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system for improving the separation process. However, to the best of the authors' present knowledge, no studies have been made on the phase diagram of the ternary system.[8] The objective of this research is to investigate and generate the phase diagrams of the ternary system at (283.15, 303.15, and 323.15) K by Schreinemaker's wet residue method[9−17] and demonstrate the temperature dependence of the ternary phase diagram.

In a ternary system involving at least one solid and one liquid phase, the composition of the solid phase is often determined indirectly, to avoid separation crystals and complete removal of the adhering mother liquor from them. Extrapolation is made by Schreinemakers' method of wet residues, based on the following: The tie line joining the composition of the pure solid and the saturated liquid in equilibrium with it is the locus of all intermediate compositions corresponding to varying amounts of solid and liquid phase. This includes the composition of the liquid phase and crystals wet with mother liquor. A straight line drawn through a pair of points representing such compositions on a phase diagram is a segment of the tie line, and therefore passes through the composition of the pure solid. The lines drawn through several such pairs of composition, each corresponding to a different original mixture, have a common intersection at the composition of the pure solid phase. The composition of the common intersection is in agreement with direct analysis obtained by crystallization.

MATERIALS SECTION

Sodium 3-nitrobenzenesulfonate was provided by Shanghai Reagent Factory (China), with a mass fraction of 0.995, and used without further purification. Sodium 4-nitrobenzenesulfonate was purchased from YuanCheng Chemical Co. Ltd. (China), with a mass fraction of 0.995, and used without further purification. The water used to prepare solutions was distilled twice (conductivity <5 $\mu S \cdot cm^{-1}$).

Apparatus and Procedure. Schreinmaker's wet residue method was used in this experiment. An external thermostat was employed in this experiment, with a device for rotating several bottles at a time. A known mass of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate was dissolved

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in 25 mL of water. The saturated solution was transferred to a conical flask. The conical flask was covered with rubber cap and placed in a device rotating the flasks at (283.15 ± 0.01 K), (303.15 ± 0.01 K), or (323.15 ± 0.01 K). The components were taken in such proportions that the composition of the resulting saturated solution fell in the desired portion of the solubility curve. Solubilities were determined by equilibrating the solute with solvent in a water-jacketed vessel with magnetic stirring in a constant temperature water bath for at least 2 days. The temperature is controlled by a constant-temperature water bath (Neslab, model RTE-101) recirculated through a copper coil in the water bath. The actual temperature in the water bath was monitored by a resistance thermometer (type, TES1300; accuracy, ± 0.01 K). Attainment of equilibrium was verified by both repetitive measurements of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate after a minimum of 2 additional days and approaching equilibrium from supersaturation by preequilibrating the solutions at a higher temperature. When the composition of the liquid phase became constant, this was taken to indicate that equilibration had been attained. The results showed that in both cases about 21 h was sufficient to reach equilibrium. After equilibrium was achieved, the liquid phase and the solid phase with a little of saturated liquid were taken out and then analyzed quantitatively. This procedure was repeated by varying the ratio of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate to obtain different compositions of the solid and liquid phases.

Analysis. Aliquots of saturated sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate solutions are transferred through a coarse filter into a tarred volumetric flask. The concentration of 3- or/and sodium 4-nitrobenzenesulfonate in water is determined using a Shimadzu-6A high-performance liquid phase chromatograph (HPLC). The Diamonsil C18 (150 mm × 4.6 mm) chromatographic column is used. The mobile phase consists of three eluents which are water, Na_2SO_4, and H_3PO_4. The uncertainty of the measurement is less than 0.01 g of sodium 3-nitrobenzenesulfonate or sodium 4-nitrobenzenesulfonate per 100 g of water. The densities (ρ) of the equilibrium liquid phase are measured using a pycnometer (11-FY) calibrated by the floating force of air with a precision of ± 0.2 mg. Each analysis was repeated three times, and the average value of three measurements was considered as the final value of the analysis (precision: ± 0.1 %).[18,19]

RESULTS AND DISCUSSION

The measured solubility and the density of the liquid phase for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system at (283.15, 303.15, and 323.15) K are shown in Tables 1, 2, and 3, respectively. The ternary phase diagrams are given in Figures 1, 2, and 3.

In the phase diagrams as shown in Figures 1, 2, and 3, W, M, and N represent water, sodium 3-nitrobenzenesulfonate, and sodium 4-nitrobenzenesulfonate, respectively. E_1, E_2, and E_3 represent the solubility of sodium 3-nitrobenzenesulfonate in water at (283.15, 303.15, and 323.15) K; S_1, S_2, and S_3 represent the solubility of sodium 4-nitrobenzenesulfonate in water at (283.15, 303.15, and 323.15) K. It can be seen from Figures 1 to 3 that along the solubility curve E_1C_1, E_2C_2, or E_3C_3, linking the component points of the liquid phase and wet solid phase and extended, the point of intersection of these tie-lines is the approximate solid-phase component for the compound sodium 3-nitrobenzenesulfonate on a wet basis. Similarly, along the solubility curve S_1C_1, S_2C_2, or S_3C_4, linking the component points of the liquid phase and wet solid phase and extended, the point of intersection of these tie-lines is the approximate solid-phase component for sodium 4-nitrobenzenesulfonate. However, at 323.15 K, along the solubility curve C_3C_4, linking the component points of the liquid phase and wet solid phase and extended, the point of intersection of these tie-lines is the approximate solid-phase component for adduct of sodium

Table 1. **Mass Fraction Solubility of the Ternary Sodium 4-Nitrobenzenesulfonate (1) + Sodium 3-Nitrobenzenesulfonate (2) + Water (3) System at 283.15 K**[a]

liquid phase		wet solid phase		density of liquid phase	
100 w_1	100 w_2	100 w_1	100 w_2	g·mL^{-1}	solid phase
0	19.13	0	81.12	1.0532	M
2.51	18.79	1.57	50.66	1.0700	M
5.26	18.70	3.59	45.12	1.1046	M
6.77	18.75	4.11	50.37	1.1361	M
7.12	18.79	42.34	50.86	1.1688	M + N
7.32	18.31	40.86	11.76	1.1642	N
7.67	16.02	27.77	12.69	1.1109	N
8.32	13.27	56.88	6.52	1.0719	N
9.49	9.08	27.45	7.28	1.0602	N
10.97	5.31	51.31	3.11	1.0550	N
12.31	2.93	51.59	1.82	1.0503	N
14.17	0	78.86	0	1.0486	N

[a] w, mass fraction; M, sodium 3-nitrobenzenesulfonate; N, sodium 4-nitrobenzenesulfonate.

Table 2. **Mass Fraction Solubility of the Ternary Sodium 4-Nitrobenzenesulfonate (1) + Sodium 3-Nitrobenzenesulfonate (2) + Water (3) System at 303.15 K**[a]

liquid phase		wet solid phase		density of liquid phase	
100 w_1	100 w_2	100 w_1	100 w_2	g·mL^{-1}	solid phase
0.00	26.51	0.00	82.44	1.0823	M
1.98	26.07	1.24	56.20	1.0854	M
4.29	25.62	2.88	53.16	1.0903	M
6.66	25.37	3.74	59.04	1.0958	M
8.97	25.28	6.88	44.07	1.1033	M
11.51	25.34	6.38	59.71	1.1125	M
13.96	25.58	8.13	56.85	1.1245	M
15.74	25.84	28.61	46.84	1.1435	M + N
16.42	22.79	37.47	17.21	1.1334	N
17.38	19.52	44.47	13.43	1.1250	N
18.34	16.25	56.21	9.03	1.1154	N
19.24	12.75	45.12	8.87	1.1122	N
20.42	9.93	42.83	7.16	1.1083	N
21.84	7.16	53.38	4.34	1.1041	N
23.25	4.57	51.04	3.16	1.0991	N
24.60	2.25	57.88	1.63	1.0963	N
26.62	0.00	81.24	0.00	1.0942	N

[a] w, mass fraction; M, sodium 3-nitrobenzenesulfonate; N, sodium 4-nitrobenzenesulfonate.

Table 3. **Mass Fraction Solubility of the Ternary Sodium 4-Nitrobenzenesulfonate (1) + Sodium 3-Nitrobenzenesulfonate (2) + Water (3) System at 323.15 K**[a]

liquid phase		wet solid phase		density of liquid phase	
100 w_1	100 w_2	100 w_1	100 w_2	g·mL^{-1}	solid phase
0	41.00	0	81.64	1.1086	M
2.07	39.24	1.14	66.38	1.1245	M
3.46	38.42	2.18	61.47	1.1442	M
6.26	37.24	2.46	75.25	1.1902	M
6.91	37.23	16.03	61.19	1.2258	M + A
9.69	36.88	31.36	43.75	1.2171	A
16.46	36.43	35.96	44.06	1.2876	A
24.50	34.71	34.18	40.22	1.3366	A
30.73	31.16	38.27	38.48	1.3710	A
33.20	27.30	41.32	38.19	1.3714	A
33.90	25.89	50.27	31.78	1.3755	N + A
34.02	25.20	62.93	14.09	1.3001	N
34.71	20.69	65.37	11.11	1.2452	N
35.72	15.52	53.85	11.05	1.2157	N
37.68	8.91	62.04	5.49	1.1807	N
39.55	4.50	70.73	2.41	1.1650	N
41.37	0	88.51	0	1.1574	N

[a] w, mass fraction; M, sodium 3-nitrobenzenesulfonate; N, sodium 4-nitrobenzenesulfonate; A, adduct of sodium 3-nitrobenzenesulfonate with sodium 4-nitrobenzenesulfonate, in which the mole ratio of the two compositions is 1:1.

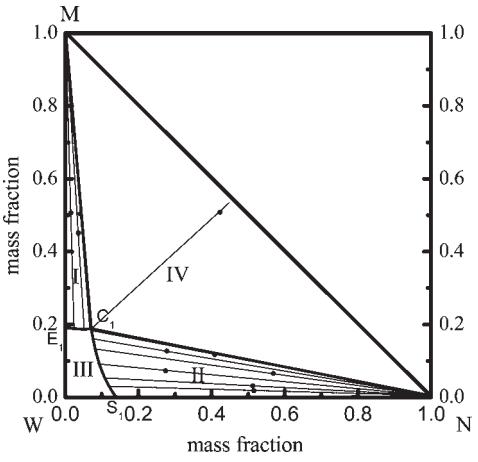

Figure 1. Phase diagram for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system at 283.15 K; W, H_2O; M, sodium 3-nitrobenzenesulfonate; N, sodium 4-nitrobenzenesulfonate; C_1, cosaturated point of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate; E_1, solubility of sodium 3-nitrobenzenesulfonate in water; S_1, solubility of sodium 4-nitrobenzenesulfonate in water.

3-nitrobenzenesulfonate with sodium 4-nitrobenzenesulfonate, in which the mole ratio of the two compositions is 1:1, named as adduct A.

E_1C_1, E_2C_2, and E_3C_3 are saturation curves corresponding to the solid-phase sodium 3-nitrobenzenesulfonate at (283.15, 303.15, and 323.15) K, respectively; S_1C_1, S_2C_2, and S_3C_4 are saturation curves corresponding to the solid-phase sodium

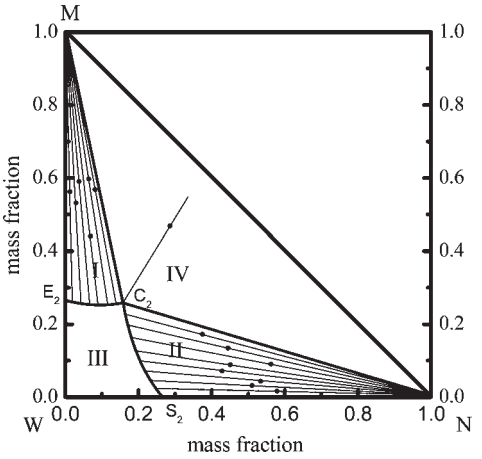

Figure 2. Phase diagram for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system at 303.15 K; W, H_2O; M, sodium 3-nitrobenzenesulfonate; N, sodium 4-nitrobenzenesulfonate; C_2, cosaturated point of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate; E_2, solubility of sodium 3-nitrobenzenesulfonate in water; S_2, solubility of sodium 4-nitrobenzenesulfonate in water.

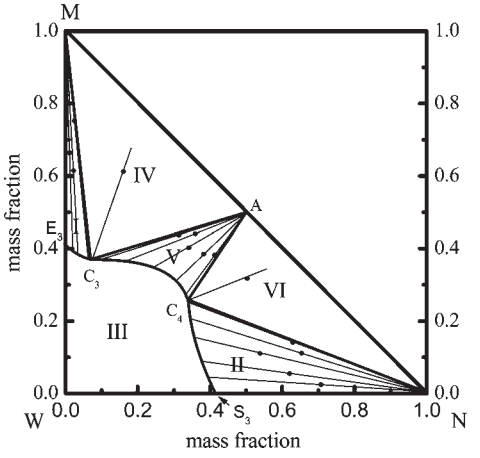

Figure 3. Phase diagram for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system at 323.15 K; A, adduct of sodium 3-nitrobenzenesulfonate with sodium 4-nitrobenzenesulfonate, in which the mole ratio of the two compositions is 1:1; C_3, cosaturated point of sodium 3-nitrobenzenesulfonate and adduct A; C_4, cosaturated point of sodium 4-nitrobenzenesulfonate and adduct A; E_3, solubility of sodium 3-nitrobenzenesulfonate in water; S_3, solubility of sodium 4-nitrobenzenesulfonate in water; W, N, and M have the same meaning as described in Figure 1.

4-nitrobenzenesulfonate; C_3C_4 is saturation curves corresponding to the solid-phase of adduct A. C_1 and C_2 are invariant points at 283.15 K and 303.15K, which represent the cosaturated solution of the solid phases sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate. C_3 is invariant point at 323.15 K, which represent the cosaturated solution of the solid phases sodium 3-nitrobenzenesulfonate and adduct A, and C_4, the cosaturated solution of the solid phases sodium 4-nitrobenzenesulfonate and adduct A.

In the phase diagrams as shown in Figures 1 and 2, there are four crystallization fields: solid sodium 3-nitrobenzenesulfonate

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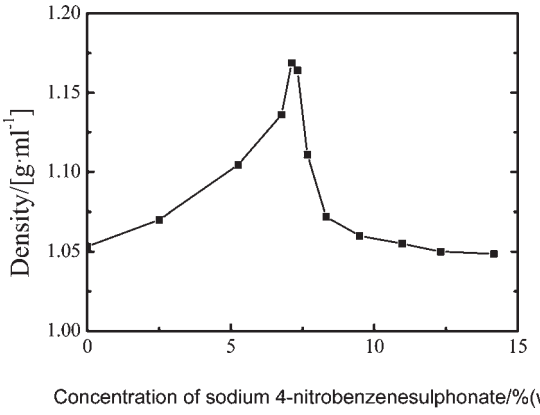

Figure 4. Density value−composition relationship diagram for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system at 283.15 K: ■, experimental data point; —, experimental relationship diagram.

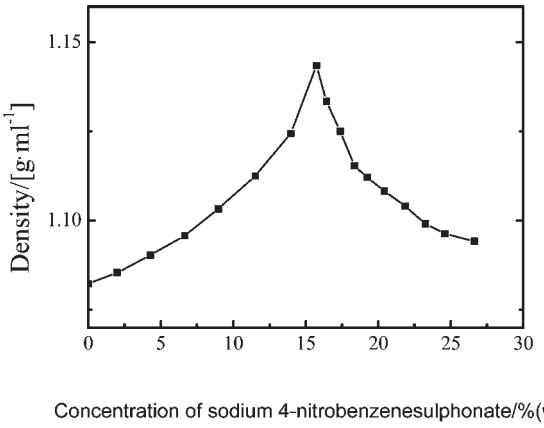

Figure 5. Density value−composition relationship diagram for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system at 303.15 K: ■, experimental data point; —, experimental relationship diagram.

(E_1MC_1 in Figure 1, E_2MC_2 in Figure 2), corresponding to the coexistence of solid sodium 3-nitrobenzenesulfonate and the saturated solution; sodium 4-nitrobenzenesulfonate (S_1NC_1 in Figure 1 and S_2NC_2 in Figure 2), the region corresponding to the coexistence of solid sodium 4-nitrobenzenesulfonate and the saturated solution; mixture of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate (NMC_1 in Figure 1, and NMC_1 in Figure 2), the region corresponding to the coexistence of solids sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate and the saturated solution; and the unsaturated region ($E_1WS_1C_1$ in Figure 1, and $E_2WS_2C_2$ in Figure 2). The phase diagram has two invariant curves at each temperature.

The phase diagram of the ternary system sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water at 323.15 K is divided into six regions by three solubility curves. The regions in the phase diagram are denoted as follows: I (E_3MC_3), crystalline region of solid sodium 3-nitrobenzenesulfonate; II (S_3NC_4), crystalline region of solid sodium 4-nitrobenzenesulfonate; III ($WE_3C_3C_4S_3$), unsaturated region; IV (AMC_3), crystalline region of solid sodium 3-nitrobenzenesulfonate and adduct A; V (C_3AC_4), crystalline region of solid adduct A; VI

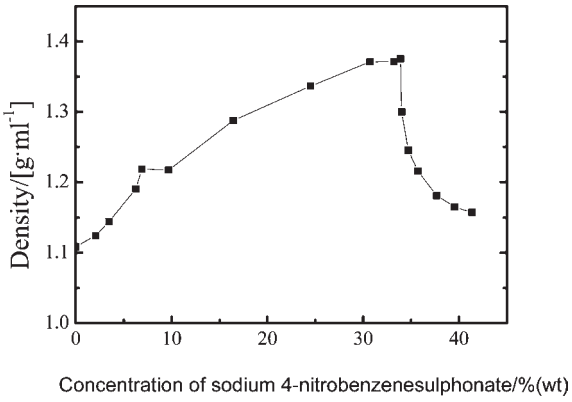

Figure 6. Density value−composition relationship diagram for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system at 323.15 K: ■, experimental data point; —, experimental relationship diagram.

(NAC_4), crystalline region of solid sodium 4-nitrobenzenesulfonate and adduct A.

Figures 1 to 3 further illustrate the temperature dependence of the phase diagram for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system. When the temperature increases from (283.15 to 323.15) K, the solubility of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate in water increases significantly, and the invariant point moves upward. The phase diagrams at 283.15 K is similar with that at 303.15 K, but different with that at 323.15 K, at which temperature the adduct A formed in the ternary system.

On the basis of data collected in Tables 1 to 3, the relationship between the density of the equilibrium liquid phase and the sodium 4-nitrobenzenesulfonate concentration values expressed in mass fraction (Figures 4, 5, and 6) is found. The density of equilibrium liquid phase depends on the total content of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate. Figures 4 to 6 illustrate that the minimum density point corresponds to the lowest total content of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate, and vice versa. The inflection point in Figures 4 and 5 correspond to the cosaturated point C_1 in Figure 1 and C_2 in Figure 2, and the inflection points in Figure 6 correspond to the cosaturated points C_3 and C_4 in Figure 3.

■ CONCLUSIONS

The solid−liquid phase equilibrium and the solubility data for the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system at (283.15, 303.15, and 323.15) K were determined experimentally. The solid−liquid phase diagrams were constructed. In addition, the densities of equilibrium liquid phase were obtained. The solid phase was confirmed by Schreinemaker's method of wet residues. Two solid phases were formed in the ternary sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water system at (283.15 and 303.15) K, which corresponded to sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate, while at 323.15 K, the three solids were formed, which corresponded to sodium 3-nitrobenzenesulfonate, sodium 4-nitrobenzenesulfonate, and adduct A. At a temperature of (283.15 or 303.15) K, the phase diagram has three crystallization fields, two univariant curves, and one invariant point. At a temperature of 323.15 K, the phase diagram

has four crystallization fields, three univariant curves, and two invariant points.

The solubility data and the ternary phase diagram for the system sodium 3-nitrobenzenesulfonate + sodium 4-nitrobenzenesulfonate + water at (283.15, 303.15, and 323.15) K can provide the fundamental basis for preparation of sodium 3-nitrobenzenesulfonate and sodium 4-nitrobenzenesulfonate from its isomeric mixtures. At 323.15 K, the approximate solid-phase component for the adduct of sodium 3-nitrobenzenesulfonate with sodium 4-nitrobenzenesulfonate, in which the mole ratio of the two compositions was 1:1, formed in the ternary system.

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