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A Study of the Behavior of Dextrose Sodium Chloride in Hydrochloric Acid Solutions¹

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Dextrose sodium chloride, $(C_6H_{12}O_6)_2 \cdot NaCl \cdot H_2O$ first elicited interest during the second quarter of the nineteenth century when investigators isolated it from the urine of diabetics and found it identical with a compound prepared from honey and salt. The most recent scientific investigation was carried out by Matsuura² who studied the system dextrose-sodium chloride-water at 24°. The double salt also has figured in the patent literature^{3,4,5,6} in recent years. This study describes the behavior of the double salt in hydrochloric acid solutions.

Experimental

Preliminary experiments with solutions of a crude factory preparation of the double salt in hydrochloric acid indicated that the compound was relatively unstable, the dextrose undergoing considerable decomposition. To ascertain the extent to which dextrose would decompose, several experiments were performed at 25–30° with anhydrous dextrose and hydrochloric acid. These experiments showed that in hydrochloric acid solutions up to 25% hydrogen chloride, dextrose is stable if the exposure does not exceed thirty minutes. Some decomposition is detectable in hydrochloric acid solutions of less than 25% hydrogen chloride at twenty-four hours. At twenty-four hours, however, decomposition occurs in hydrochloric acid of 25% hydrogen chloride to the extent of about 15%. In 36%

hydrochloric acid, decomposition was noticeable in thirty minutes and proceeded to the extent of 60–65% in fifteen days. These results signified that the double salt would probably remain stable in hydrochloric acid solution up to 25% concentration during a period of thirty minutes.

To carry out this study, double salt was prepared from pure anhydrous dextrose and c. p. sodium chloride. Analysis of the dry product showed the compound to contain 82.66% dextrose and 13.40% sodium chloride as compared to the calculated values of 82.49% and 13.39%, respectively. Solubility determinations proved the compound to be congruently soluble at 30°, forming a saturated solution of 61.4% anhydrous dextrose sodium chloride. The dextrose analytical values were determined by Lane and Eynon's⁷ method; those for sodium chloride, by Mohr's method.

Phases Precipitated from Aqueous Solutions of Dextrose Sodium Chloride by Hydrogen Chloride.—Ten systems, the data for which are compiled in Table I, were investigated. Each system was treated in the following manner: Dextrose sodium chloride and water were weighed into a suitable screw-cap bottle and maintained at 30° in a constant temperature bath, thermostatically controlled at $30 \pm 0.1^\circ$, for a period of not less than one-half hour. After carefully drying and weighing the bottle, the calculated volume of concentrated hydrochloric acid was added and the bottle again weighed. Immediately thereafter, the bottle was replaced in the constant temperature bath and kept there for one-half hour; the bottle and contents were shaken at least once every five minutes. At the end of the period, samples of the solution and wet residue were analyzed for their hydrogen chloride, sodium chloride and dextrose contents. In addition, a microscopic examination was made to ascertain the nature of the solid phase. The total weight of each system did not exceed 12.5 g., which provided enough sample for analytical purposes in all cases except two where wet residues were concerned.

The hydrogen chloride, sodium chloride and dextrose contents of the solutions and wet residues were determined according to the following manner: solution was filtered into a weighing bottle, by means of a filter stick and gentle suction; wet residue was transferred into another. After weighing, each was transferred quantitatively to a suitable volumetric flask and the quantity of hydrogen chloride estimated with standard sodium hydroxide and phenolphthalein. After discharging the indicator color with dilute nitric acid, the sample was diluted to the mark, aliquots removed for the sodium chloride determination, and the remainder used for the determination of dextrose. Sodium chloride was estimated by determining total chlorides by Mohr's method and subtracting the hydrogen chloride equivalent. Dextrose was evaluated by Lane and Eynon's⁷ method. Water was estimated as the difference.

The data for the first eight experiments, Table I, are graphically represented in Fig. 1, the ternary diagram for the system dextrose sodium chloride (anhydrous)-hydrogen chloride-water.⁸ The circles represent the compositions of the systems taking the sum of the dextrose and sodium chloride percentages equal to "D.S.," anhydrous dextrose sodium chloride. Point S corresponds to

(7) J. Henry Lane and Lewis Eynon, *J. Soc. Chem. Ind.*, **42**, 32T (1923).

(8) We wish to thank Dr. John E. Ricci, New York University, University Heights, New York 53, N. Y., for suggesting the method of representing the data.

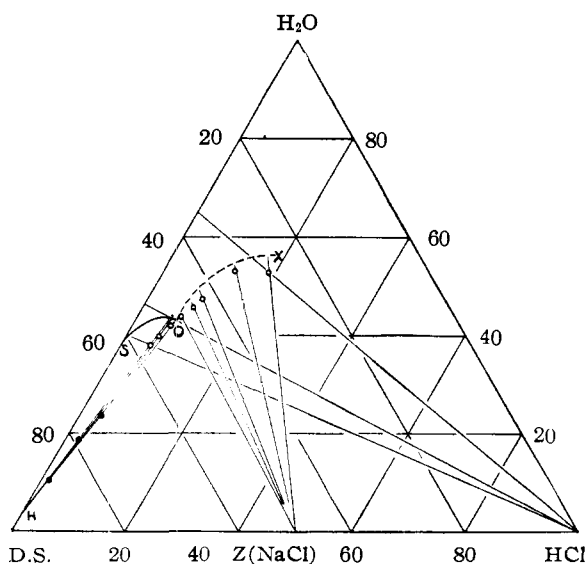


Fig. 1.—Ternary system D.S.—H₂O—HCl.

(1) This paper was presented before the Division of Sugar Chemistry and Technology of the American Chemical Society, Detroit, Michigan, September, 1940.

(2) Matsuura, *Bull. Chem. Soc. Japan*, **2**, 44 (1927).

(3) Carl Rach, U. S. Patent 1,825,603.

(4) Julian K. Dale, U. S. Patent 2,100,433.

(5) T. H. Barnard and Paul L. Stern, U. S. Patent 2,150,146.

(6) R. C. Wagner and Paul L. Stern, U. S. Patents 2,150,197 and 2,175,369.

TABLE I
 EXPERIMENTAL DATA

Expt. no.	I	II	III	IV	V	VI	VII	VIII	IX	X
% Compn. of system										
C ₆ H ₁₂ O ₆	24.5	29.4	36.9	39.1	41.5	44.0	46.6	48.9	42.8	47.2
NaCl	4.0	4.8	6.0	6.3	6.7	7.1	7.6	7.9	6.9	7.7
HCl	18.6	12.7	9.8	9.0	7.9	6.9	5.9	5.1	9.5	8.6
H ₂ O	52.9	53.1	47.3	45.6	43.9	42.0	39.9	38.1	40.8	36.5
% Compn. of solution										
C ₆ H ₁₂ O ₆	24.7	29.7	37.2	39.4	42.1	42.9	42.6	42.7	37.1	36.6
NaCl	1.4	4.0	5.5	6.2	6.7	7.4	7.4	7.2	4.5	4.4
HCl	19.3	13.5	10.1	9.2	8.1	7.3	6.7	6.1	11.4	11.6
H ₂ O	54.6	52.8	47.2	45.2	43.1	42.4	43.3	44.0	47.0	47.4
% Compn. of wet res.										
C ₆ H ₁₂ O ₆	11.8	16.1	19.7			62.7	75.8	68.0	69.1	73.6
NaCl	52.7	59.7	54.8			10.0	12.3	11.0	20.5	17.4
HCl	9.3	7.3	4.8			3.7	1.3	2.3	1.6	1.2
H ₂ O	26.2	16.9	20.7			23.6	10.6	18.7	8.8	7.8
Microscopic exam. solid phase	NaCl	NaCl	NaCl	NaCl	NaCl	Dextrose Sodium Chloride	Dextrose Sodium Chloride	Dextrose Sodium Chloride	Dextrose Sodium Chloride	Dextrose Sodium Chloride

the solubility of the compound on the anhydrous basis. Curve SO represents the compositions of solutions in equilibrium with dextrose sodium chloride hydrate, point H. Solutions O-X, resulting from systems I through V, lie just slightly off the plane of the diagram and are in equilibrium with sodium chloride, point Z, which is on the side of the plane of the diagram opposite solutions O-X. Attention must be called to the fact that the broken curve representing solutions O-X refers to points only on the solution surface of the quaternary system dextrose-sodium chloride-hydrogen chloride-water saturated with sodium chloride. Systems IX and X, Table I, show that if the percentage of hydrogen chloride is increased in systems VI and VIII, two solid phases, dextrose sodium chloride and sodium chloride, precipitate. The analytical figures in Table I for both solutions and wet residues as well as microscopic examination were used to identify the solid phases.

In conclusion the addition of hydrogen chloride to aqueous solutions of dextrose sodium chloride of greater than 53.6% anhydrous dextrose sodium chloride (found by extending the line O-HCl,

Fig. 1, to the D.S.-H₂O side) precipitates, first, the double salt, (C₆H₁₂O₆)₂·NaCl·H₂O, and secondly, double salt and sodium chloride. Addition of hydrogen chloride to aqueous solutions of double salt containing less than 53.6% anhydrous dextrose sodium chloride causes sodium chloride to precipitate.

Summary

1. The instability of dextrose in hydrochloric acid solutions at 25-30° is reported.
2. The solubility of dextrose sodium chloride on the anhydrous basis at 30° has been determined as 61.4%.
3. Phases precipitated from aqueous solutions of dextrose sodium chloride by hydrogen chloride at 30° were determined. Depending upon the dextrose sodium chloride and hydrogen chloride contents of the system, either dextrose sodium chloride, sodium chloride, or both were precipitated.

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(9) Original manuscript received November 13, 1944.