

A New Method for the Isolation of Sucrose from the Mixture of Glucose, Fructose and Sucrose.

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The processes for the isolation of pure d-glucose and d-fructose by fractional crystallisation in the glacial acetic acid solution of their mixture with or without sucrose, were described in the study of the kaki-fruit¹. The preparation of pure crystalline sucrose from such a mixture was a tedious task especially when it was found in smallest proportions compared with other sugars, notwithstanding that sucrose crystallizes from the concentrated solution with remarkable facility.

The principle from which the new method was derived, was to isolate the sucrose after separating the reducing sugars from the solution by oxidation, transforming them into insoluble salts of the sugar acids.

To attain this purpose, the oxidation of sugars in a neutral solution is preferable, since the reducing sugars, by this treatment, were all converted into sugar acids, while the sucrose remains in the solution, being unaffected by the action of the oxidizing agent.

The method for oxidizing the sugars with bromine water in presence of calcium carbonate has been investigated. The necessity of precipitating and removing the calcium gluconate and other calcium salts formed by the reaction, from the solution makes the method inconvenient.

The oxidation with the yellow oxide of mercury instead of bromine water, in presence of calcium carbonate, will be appreciated

¹ S. Komatsu and H. Ueda: *J. Biochem.*, **1**, 181 (1922); S. Komatsu and M. Ishimasa: *Ibid.*, **2**, 301 (1923).

equally with the bromine method in yielding gluconic acid and other sugar acids from glucose and fructose, and also in leaving sucrose in the solution, unaffected by the action of the reagent¹. Moreover, the employment of yellow mercuric oxide as an oxidizing agent obviates the trouble of removing calcium bromide from the solution, and consequently facilitates the isolation of sucrose found in any proportion in the sample.

A solution was made of 4 gm. sucrose and 6 gm. d-glucose in 100 c.c. of water. This mixture was then treated according to the directions suggested by A. Blanchetière² with 25 gm. yellow mercuric oxide and 10 gm. calcium carbonate. After boiling for 2 hours, the solution was filtered from the mercurous oxide and mercury formed, concentrated to small volume under reduced pressure, and the calcium salts of the acids formed by the oxidation of the reducing sugars were precipitated by the addition of alcohol. In order to precipitate completely the calcium salts only, the percentage of alcohol in the solution should be kept at 80% by volume in the solution while sucrose remains; of the sugars remaining in the solution, 36% was glucose and 64% sucrose, calculated by the reducing power of Fehling's solution before and after inversion.

A solution of 4 gm. sucrose, 3 gm. d-glucose and 3 gm. d-fructose in 100 c.c. of water, was oxidized with 25 gm. mercuric oxide and 10 gm. calcium carbonate, in the same manner as before.

The solution separated from mercury, mercurous oxide and calcium salts of organic acids, which was ascertained to contain 1.8 gm. of the reducing sugar and 2.9 gm. sucrose, (ratio 1:1.6) was concentrated under reduced pressure to a thick syrup. The syrup was poured out of the flask and mixed thoroughly with 2 volumes of hot glacial acetic acid. After cooling to room temperature, it was seeded with sucrose and set aside to crystallize in an ice-cooled chamber. The sucrose

¹ O. v. Lippmann: D. Chem. Zuckerarten (1904), 1229, 1277.

² Bull. soc. chim., (4), 35, 345 (1923).

A New Method for the Isolation of Sucrose from a Mixture of Glucose, etc. 163

crystallized in white lumps was separated from the mother liquor and washed with alcohol and ether and then dried as usual. The yield was 1.1 gm. which corresponds to about 40% of theory.

The sample recrystallized from the ethyl alcohol solution, melts at 181°, and showed the following constant-specific rotation in a 1dm. tube :

$$[\alpha]_D^{21} = \frac{22.42 \times 10}{0.3351} = +66.68$$

SUMMARY

Sucrose was isolated in crystalline state from the mixture of glucose, fructose and sucrose, after transforming the reducing sugars into the sugar acids by the oxidation of yellow oxide of mercury in presence of calcium carbonate.

It was found necessary to keep the solution in neutral to reduce the hydrolysis of sucrose.

The sucrose was crystallized from the glacial acetic acid solution as usual.

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