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# THE NATIONAL BUREAU OF STANDARDS AND THE ESTABLISHMENT OF HARD REFINED DEXTROSE AND LEVULOSE INDUSTRIES

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In the present stage of development there are three sweet carbohydrates of vital importance as food, namely, dextrose (corn sugar), sucrose (ordinary sugar), and levulose. All have about equal food value.[1] There is, however, a difference between them which is of fundamental importance. Compared with sucrose, dextrose has a lower sweetening power, whereas levulose has a higher sweetening power. Therefore, if the cost of dextrose and levulose should be the same as or less than that of hard refined sucrose, man would have available, by mixing these two sugars, a sugar of any intermediate degree of sweetness desired. The per capita consumption of sugar has increased until this commodity is now rated as one of the necessities of life; and in addition, the world demands, except in a few instances, a chemically pure product for its sweetening. Thus, it is not surprising that there is a world consumption of approximately 26 million tons of a chemically pure product annually. The United States alone consumes over six million tons, one-half of which amount it is necessary to import from foreign countries, and only a little more than one-sixth of which is produced in con-

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1. Large calories in 1 pound of sucrose, 1,794; dextrose, 1,697.8; levulose, 1,703.3.

tinental United States.

Any sugar, to be marketed at a low cost per pound, must be produced by a method which permits its being crystallized from water solution. The National Bureau of Standards began its experimentation in this field by producing in quantity the sugar-alcohol, mannite, for the first time from water solution. This was accomplished on a considerable scale for the Field Medical Hospital Service of the United States Army during the World War. Our previous supply of this indispensable substance had come from Germany, where it had been prepared by precipitation with alcohol.

DEXTROSE

Inasmuch as the world had long waited for a carbohydrate of high food value, great purity, low cost, and low sweetening power, the National Bureau of Standards' resources were next turned to the solution of the problem of producing hard refined dextrose on a commercial scale. The establishment of a large manufacturing industry presupposes the existence of an adequate supply of cheap raw material. With its enormous annual production of corn the United States had available in the form of starch an unlimited supply of a very cheap raw material suitable for the manufacture of a sugar. An exhaustive study of the crystallization of dextrose from water solution was carried out. This work resulted in the establishment of a new industry which has become of large proportions in a few years' time. Some millions of dollars had already been spent by that industry in its efforts to produce low-cost pure crystalline dextrose on a commercial scale.

In 1919, after having furnished the research department of the Corn Products Refining Company with theoretical and technical information on the subject during the course of several years, and no successful commercial production having resulted, the Bureau completed the theoretical and technical work necessary for the production of either the anhydrous or the hydrate form of the dextrose crystal at will. It then sent one of its staff members from the carbohydrate laboratory to the Edgewater, N.J., plant of the Corn Products Refining Company where he soon threw down from water solution 4,000 pounds of crystalline dextrose, and without the use of special equipment. It was thus demonstrated for the first time that it was perfectly feasible to produce chemically pure dextrose at a low cost on a large manufacturing scale. The Bureau continued the experimental work on a commercial scale, sending the above-mentioned member of its staff to the Edgewater plant where experiments were made. The possibility of commercial production of hard refined dextrose was so completely demonstrated by these experiments that the Bureau was requested to design a semicommercial plant costing approximately $250,000. The purpose of this plant was to determine whether any unexpected difficulties would develop in the application of the process to large-scale production. The design of such an experimental plant was undertaken and by the time the drawings were completed it was apparent that the Bureau's process was applicable to large-scale production. The Corn Products Refining Company immediately started work on a 1 1/2 million-dollar factory in connection with its Argo, Illinois plant. This plant was put into successful operation within a comparatively short time. Other factories have been built and are in operation in this country and in a number of foreign countries. In addition, a large hard refined dextrose plant producing the anhydrous form of the sugar exclusively has been built and is in operation in North Kansas City, Mo. As a result there has developed a constantly expanding demand for hard refined anhydrous dextrose, especially for use as a raw material in the manufacture of medicinals and other products.

As a result of the Bureau's work, other corn products refining companies, namely, Penick & Ford, Cedar Rapids, Iowa; Clinton Company, Clinton, Iowa; and the American Maize Products Co., Roby, Indiana, are producing hard refined dextrose. Dextrose has been vitally needed as an adjunct to the use of ordinary sugar. Satisfactory production in some large-scale industries could not be made because of the lack of such a product. It was needed as the perfect carbohydrate "filler". It is also needed in the wine industries of Europe, and the Corn Products Refining Company now has a large factory in Germany. All of this means an increased consumption of American corn, and since this tends to stabilize the price of cash corn, it is difficult to overestimate the value of the hard refined dextrose industry to the American farmer. This increased consumption has an importance much greater than the quantity involved because it is the excess crop which must be sold in foreign markets that unfortunately fixes the price of the entire crop. The dextrose industry gives promise of being a most important factor in the domestic disposition of the average excess corn production. Inevitably the dextrose industry will provide one of the most dependable outlets for our surplus corn. In appearance dextrose cannot readily be distinguished from ordinary granulated sugar and is on the market in three principal sizes of grain, namely, coarse, fine, and standard granulated.

A bushel of corn (60 pounds) produces approximately 25 to 27 pounds of dextrose; 5 pounds of hydrol (molasses); one pound of oil; and 27 pounds of feed.

One of the attributes of the crude dextrose (corn sugar) of commerce which originally interested the National Bureau of Standards was its low sweetening power. With its commercial production in the form of a granulated sugar it has been found necessary to revise existing ideas in regard to its relative sweetening power. If we arbitrarily give sucrose a rating of 100 on the basis of its sweetening power, the best scientific study of the relative sweetening power of the common sugars gives dextrose 74.3, sucrose 100, and levulose 173.3. Obviously such data if applied to a specific individual are necessarily approximate. While dextrose crystallizes in two different crystalline forms, the anhydrous and the hydrate, the latter having 9 percent water of crystallization, it is the hydrate form of the hard refined sugar which is most largely produced commercially. Although accurate figures are not available, production in continental United States now amounts to several hundred thousand tons annually. The sugar is perfectly stable with excellent keeping qualities. Theoretically it has an osmotic pressure higher than that of sucrose and should therefore exert a greater preserving action. The commercial uses to which hard refined dextrose is applicable are already extensive and are steadily increasing in number. Of these it is of interest to mention that the making of this sugar widely available commercially in pure form has resulted in its extensive use in medicine, and especially in its use as a food in children's hospitals.

## LEVULOSE

After the results with dextrose had been obtained and a large new world industry created, the National Bureau of Standards felt justified in turning its resources to an experimental study of the last of the three sugars mentioned above, namely, levulose. If it could be proved that the large-scale commercial production of hard refined levulose was practicable and feasible from an economic standpoint, some of the land now used for growing wheat would be utilized for growing tubers to produce levulose, and this would greatly lessen the importance and magnitude of the surplus wheat problem of the continental United States. It is this possibility that has been the principal actuating motive back of the Bureau's investigations. Such a solution would be an ideal one for surplus crops.

The economic and commercial possibilities of levulose, could it be successfully crystallized from water solution, have long been given serious attention in Europe as well as in America. The creation of a large-scale continuous-flow process for the commercial production of hard refined levulose is, however, far more difficult than the similar problem encountered in creating the hard refined dextrose industry. In the case of dextrose there was an extensive scientific literature. In numerous instances the history of these experiments was of importance in the Bureau's prosecution of the dextrose problem. Lines of attack that had proved to be unsuccessful served as guide posts and thereby was eliminated much work which would have resulted from their repetition. In the case of levulose, however, although this sugar has been known scientifically for nearly a century, the literature in relation to its production was found to be practically nil. The Bureau's first publication on this subject was presented before the Ithaca, N.Y. meeting of the American Chemical Society the week of September 8, 1924, and carried the announcement that the National Bureau of Standards had developed a laboratory method for the successful crystallization of levulose from water solution. It is perhaps permissible to state that no development has occurred in many years which exhibits such potentialities for profoundly influencing man's food supply. The production of sugar is one of the world's largest industries. A new industry which gives promise of modifying the production, or even the distribution, of sugar is a thing of first importance to mankind.

Levulose is not only the sweetest; it is also the most soluble of all the sugars. It has long been scientifically the most fascinating and elusive. The human race consumes large quantities of it and of dextrose in honey and in sirup. Physiologically it is probably as easily assimilable as dextrose. Small amounts have been made by special chemicals companies (by the use of alcohol) for scientific purposes, and also for use in dietetics. When ordinary sugar is eaten it is broken down into dextrose and levulose in order that it may become assimilable. Dextrose and levulose undoubtedly constituted the principal sugars upon which man developed as an animal. They are predigested. Sucrose is a new thing which has been added with the development of civilization. The first commercial production occurred only about six hundred years ago. Levulose corresponding in purity to that of hard refined dextrose or sucrose has always been so costly that it could be used only for scientific purposes. The Bureau has succeeded in throwing down pure crystalline levulose from water solution. So far as their general appearance is concerned it is difficult to distinguish between the sugars levulose, dextrose, and sucrose in the hard refined or granulated form. The crystals of levulose are, however, more brilliant than those of any other sugar.

As a source of raw material for the commercial production of levulose the National Bureau of Standards has utilized most extensively the tuber of the Jerusalem artichoke. This plant is a native American weed which seems to survive the attacks of its enemies, grows well anywhere in the United States, produces from 8 to 20 tons to the acre, and contains polysaccharides which upon conversion yield from 10 to 16 percent of levulose, estimated upon the weight of the tuber. It is cultivated about the same as corn - three times with the cultivator if possible. Through the generosity of the Honorable Joseph C. Sibley, former member of Congress from Pennsylvania, 1,000 bushels of Mammoth

French White Jerusalem artichoke tubers were made available for the Bureau's use. It was with this material that the first experiments on commercial levulose production were carried out. In addition, the availability of the carbohydrate, inulin, in the dahlia tuber was studied as a possible source of raw material. It was found that this tuber is probably intrinsically superior to the artichoke tuber as a source of levulose. Our experimental work showed that it possessed less objectionable characteristics, especially impurities. However, no experiments have ever been made on the commercial growing of dahlia tubers for the purpose of producing the largest possible tonnage of sugar per acre. It is entirely conceivable, as we have frequently pointed out, that a dahlia tuber, high in inulin, hardy, and giving large tonnages per acre might be developed. If this were done, it would in our opinion certainly be preferable to the artichoke tuber as a source of raw material.

Approximately 200 pounds of hard refined levulose was produced during the Bureau's first crude experiments to discover a method suitable for large-scale production. The far more difficult problem of determining whether a continuous-flow process suitable for commercial production was feasible was then undertaken. A pilot plant designed to produce in excess of a half ton of hard refined levulose daily was developed. This was a large undertaking owing to the fact that practically all equipment had to be especially designed and built. Very little commercial equipment was available or suitable for the purpose at hand. This work was continued for a number of years and a semicommercial-scale factory developed at this Bureau was practically completed. As a result of the Bureau's work, commercial production of hard refined levulose seems inevitable, but whether or not an important industry is developed depends upon whether a production method can be developed which will lower the cost per pound to a point where the sugar can be used on a large scale.

This Bureau's work toward the creation of a levulose industry has been admirably supplemented by work of the Bureau of Plant Industry of the Department of Agriculture in developing improved types of tubers. The artichoke tuber seems to offer very great promise of selection to the end that a tuber can be developed which will give a greatly increased yield of sugar. There are thousands of varieties of the tuber growing wild on the American continent. Several hundred varieties have been collected and studied by the Bureau of Plant Industry. They have published Technical Bulletin No. 33, "The Jerusalem Artichoke as a Crop Plant", Technical Bulletin No. 514, "Studies of the Culture and Certain Varieties of the Jerusalem Artichoke", and Leaflet No. 116, "Growing the Jerusalem Artichoke"

A levulose industry to be of real value and a permanent success must be founded upon a process which will produce the hard refined sugar at a price to permit it to be readily sold. It is not a question of merely producing levulose. Everyone has been able to do that since our first successful experiments in 1924 in crystallizing the sugar from water solution. We have repeatedly received offers from responsible parties and experienced sugar people to enter levulose production. This we have declined to do until we felt that we had the data to make a large-scale industry possible.

This Bureau had practically completed its semifactory-scale plant for the production of levulose when the necessity for Government economy curtailed its funds in 1933. Prior to that time we had been greatly handicapped by the lack of suitable extraction equipment, which was essential to the securing of liquors of sufficient purity to permit of successful operation. We had been forced to use a hydraulic press for the extraction of the juice. The extraction requires a diffusion process similar to that used in the beet sugar industry. We finally succeeded in designing and having constructed a special diffusion battery capable of performing the work and had in installed, when the curtailment of further experimentation came. Of course it is not to be understood from this that the plant was perfected. Additional experimentation would undoubtedly develop the necessity for further modification. But we had developed for the first time all the essential units for the first approximation to a complete commercial experiment. It would probably require two years of additional experimentation to obtain with the Bureau's semifactory-scale plant the additional scientific and engineering data needed for the design of a continuous-flow large-scale commercial plant, if such plant be possible, and to obtain approximate data on the cost of producing a pound of the refined sugar when operating on a large commercial scale.

The following publications dealing with the Bureau's investigations of the scientific and technical problems involved in the development of a method for the commercial production of hard refined levulose have been published:

| Series | Price | Title |
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| S519 | 10¢ | The preparation of levulose. Richard F. Jackson, Clara Gillis Silsbee, and Max J. Proffitt. BS Sci. Pap. 20, 587 (1926). |
| RP79 | 5¢ | A crystalline difructose anhydride from hydrolyzed inulin. Richard F. Jackson and Sylvia M. Goergen. BS J. Research 3, 27 (1929). |
| RP224 | 5¢ | Note on the individualities of anhydrofructose and difructose anhydride. Richard F. Jackson and Sylvia M. Goergen. BS J. Research 5, 733 (1930). |
| RP251 | 5¢ | The constant occurrence of nonreducing disaccharides in hydrolyzed inulin. Richard F. Jackson and Emma McDonald. BS J. Research 5, 1151 (1930). |
| RP299 | 5¢ | Two new crystalline difructose anhydrides from hydrolyzed inulin. Richard F. Jackson and Emma McDonald. BS J. Research 6, 709 (1931). |
| RP426 | 10¢ | Some physical properties of levulose and its estimation by copper reduction methods. Richard F. Jackson and Joseph A. Mathews. BS J. Research 8, 403 (1932). |
| RP495 | 5¢ | Analytical methods for the determination of levulose in crude products. Richard F. Jackson, Joseph A. Mathews, and W.D. Chase. BS J. Research 9, 597 (1932). |
| RP611 | 5¢ | The stability of levulose in aqueous solutions of varying pH. Joseph A. Mathews and Richard F. Jackson. BS J. Research 11, 619 (1933). |
| RP832 | 5¢ | Yield and purity of levulose derived from the calcium levulate process. Richard F. Jackson and Joseph A. Mathews. J. Research NBS 15, 341 (1935). |
| RP840 | 5¢ | Design and construction of an experimental diffusion battery. Max J. Proffitt. J. Research NBS 15, 441 (1935). |
| RP931 | 5¢ | Dimensions of Jerusalem artichoke cossettes. Max J. Proffitt, John A. Bogan, and Richard F. Jackson. J. Research NBS 17, 615 (1936). |
| | | The structure of difructose anhydride III. Emma McDonald and Richard F. Jackson. (Not yet published. Presented before the Division of Sugar Chemistry, American Chemical Society, at Chapel Hill, N.C., April 12-15, 1937.) |

With the exception of the last paper, the above publications may be secured from the Superintendent of Documents, Government Printing Office, Washington, D.C., at the prices indicated.