

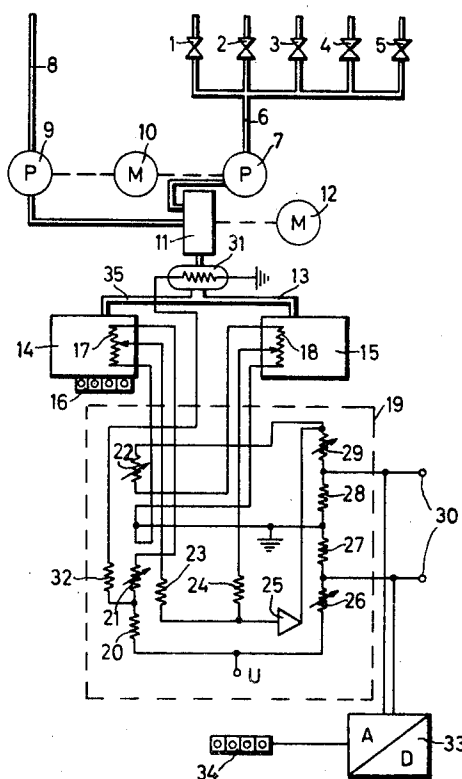
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[54] **METHOD AND APPARATUS FOR MEASURING
THE PURITY RATIO OF INTERMEDIATE
PRODUCTS IN SUGAR MANUFACTURE**
17 Claims, 2 Drawing Figs.

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[56] **References Cited**
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ABSTRACT: The proportion of sucrose in the whole of the dissolved substances of intermediate products in sugar manufacture is measured by: measuring the refractive index n of a sample of an intermediate product, measuring the polarimetric rotation α of the same sample and then combining these measurements in the relationship $\alpha/n - n_0$, to produce a "purity quotient" (n_0 being the refractive index of pure water.) The numerical value of this quotient varies with the temperature of the sample. Therefore the temperature of the sample is maintained constant within a certain range and an additional temperature correction is effected. In a preferred form of apparatus for performing the measuring method, the resultant of the calculation of the quotient is reproduced in both analog and digital form respectively for controlling automatic process control computers and for actuating a device for indicating the measurements in visible form.



METHOD AND APPARATUS FOR MEASURING THE PURITY RATIO OF INTERMEDIATE PRODUCTS IN SUGAR MANUFACTURE

The present invention relates to a method and apparatus for the automatic continuous measurement of the purity ratio of intermediate products in sugar manufacturing.

In the manufacture of sugar, crude juice is first obtained from the raw material. In the case of sugar beets this is done by extraction from washed and chipped beets with hot water. In the case of sugarcane, the juice is squeezed out by roll crushers.

The crude juice thus obtained is purified—for example, by means of the lime-carbon dioxide method—to remove a major portion of the nonsugar substances. The resulting thin juice is evaporated in an evaporator station to a thick juice, which contains about 60 to 65 percent dry substance.

The thick juice flows into the sugarhouse where it is boiled to massecuite, which is a thick mixture of light crystals and dark syrup. The massecuite is cooled in mash troughs and when centrifuged. The mother syrup which is separated by this step is called runoff.

After this separation, and particularly in the production of white sugar, a washing medium (for example, water) is sprayed on the sugar layer in the centrifuge to remove the syrup particles adhering to the sugar crystals. In this process sugar is also dissolved and the wash syrup resulting from a repeated centrifugation has a high sugar content. The centrifuged wash syrup is thereafter further concentrated by boiling.

The runoff, or mother syrup from the boiled and centrifuged massecuite, is again boiled, cooled, centrifuged, and washed in a successive stage.

Depending on the type of factory (raw sugar or white sugar factory), the crystallization and the separation of the sugar are effected in at least two or three successive stages. Runoffs and wash syrups are recycled through the various boiling and centrifuging stages in various ways until molasses, which is a runoff that can no longer be crystallized, is obtained.

For an economical manufacture of sugar it is important to keep the power consumption in the various boiling stages at a minimum and to control the process so that molasses with a low sucrose content is obtained. To this end it is necessary to measure the various intermediate products (runoffs, wash syrups, and charges of the various boiling stages), giving particular attention to the proportion of pure sucrose in the total of nonhydrous substances. This proportion in percentages is indicated by the so-called purity quotient, which is frequently called the "quotient" for short.

The purity quotient is usually written in the form (Pol/Brix)·100, where Pol denotes the sucrose concentration in degrees S, determined polarimetrically according to the ICUMSA method (International Commission for Uniform Methods of Sugar Analysis). Brix denotes the refractometrically determined total dry substance content in degrees Brix according to the international sugar scale in the version of 1936, which is still used today.

Since the knowledge of the purity quotient is of paramount importance for the processing of the intermediate products, the determinations of the quotient represent a major part of the work done in the laboratory of a sugar factory.

The expenditure in time and energy for the determination of the quotient is relatively great, because the material to be measured requires certain preparations. Since the quantities degrees S and degrees Brix are not equidimensional, the temperature for the measurement is fixed, according to definition, at 20° C. For measuring the quantity degrees Brix, the sample material is diluted in the proportion 1:1 before measurement, then measured at 20° C., and the result is then multiplied by the factor 2. For measuring the quantity degrees S, 26 grams of the sample material are diluted to make 100 ml. of solution are then precipitated with lead acetate, then the solution is filtered and measured polarimetrically at 20° C.

Due to the preparatory operations the time required for a quotient determination is about 20 minutes. Moreover, this

conventional method of quotient determination cannot be fully automated.

Efficient sugar production requires that the proportion of sucrose in the total of nonhydrous substances—that is, the purity quotient—in various intermediate products be measured as rapidly, accurately and automatically as possible. But, improvement in this regard is not possible using the conventional method of quotient determination.

It is, therefore, an important object of this invention to provide an improved method for measuring the purity ratio of the intermediate products in the sugar manufacture, and to provide such a method which is rapid, accurate and which is adapted to being performed automatically.

According to the invention, the angle of rotation α of a sample liquid is determined polarimetrically and the refractive index n of the same sample liquid is determined refractometrically. From these quantities, and from the known refractive index n_0 of pure water, is then formed the quotient $\alpha/(n-n_0)$ which defines the proportion of sucrose in the total of nonhydrous substances.

The invention will now be described in detail with reference to the accompanying drawings in which:

FIG. 1 is a graph in which the specific rotations α are plotted against the refractive indices n for four samples, each having a different sucrose content, and

FIG. 2 is a schematic diagram of apparatus for measuring the purity quotient in accordance with the invention.

As can be gathered from the graph FIG. 1 every sample yields a straight line whose slope is characteristic of the purity of the sample. All these lines start from the value n_0 , as the specific rotation α is zero at sucrose concentration zero, while the refractive index n has the value n_0 , n_0 being the refractive index of pure water. This means that the purity can, indeed, be described by the new expression $\alpha/(n-n_0)$.

The expression $\alpha/(n-n_0)$ is free of concentration-dependent coefficients of measure and is, therefore, independent of the concentration of the sample. α , which is the function of the difference between two refractive indices, namely the indices of the levorotatory and of the dextrorotatory polarized light, thus has a different temperature dependence than the pure refractive index n for nonpolarized light. It is, therefore, necessary to relate the determination of the quotient $\alpha/(n-n_0)$ to a constant temperature. This temperature can, however, be selected at random, in contrast to the conventional quotient determination which is only exact at 20° C. In the case of temperature deviations from the selected reference value, it is possible to make the correction by simple mathematics, or by an automatic correction device in the measuring apparatus.

Since the sample for the measurement of the quotient $\alpha/(n-n_0)$ does not require special preparation, and since the values for α and n can be measured rapidly and with great accuracy, the quotient can likewise be measured rapidly and with great accuracy. The method of this invention thus contributes to the efficiency of the conventional discontinuous sugar-manufacturing process and in addition provides the basis for an automated, or partly automated continuous manufacturing method.

The quotient $\alpha/(n-n_0)$ can be related in a simple manner to the purity quotient Pol/Brix·100; it is only necessary to multiply the quotient $\alpha/(n-n_0)$ by a factor F, which takes into account the polarimetric path length, the measuring temperature, and other quantities. The factor F can be determined mathematically, but empirical determination by measuring a pure sucrose solution, which has according to definition a purity quotient of 100 is preferable. The information content is however, not increased by relating the quotient $\alpha/(n-n_0)$ to the purity quotient (Pol/Brix)·100. The information gain apparently achieved by said relating is the possibility to make a direct comparison with the values obtained by the conventional method. This is not necessary for factory control.

In a preferred manner of using the method of this invention the quotient $\alpha/(n-n_0)$ is obtained by translating the angle of rotation α and the refractive index n into electric quantities

and forming the quotient $\alpha/Cn-n_0$ electrically from these quantities and from a corresponding input value for n_0 . Also, the quotient thus determined can be multiplied electrically by the proportionality factor F.

The quotient $\alpha/(n-n_0)$ can be used, before or after multiplication by the factor F, for the automatic regulation or control of the sugar-manufacturing process. It is advantageous to produce said quotient in both analog and digital form, the analog form being particularly suited to being fed directly into a process computer for automatic operation control. The digital form is particularly suited for actuating an indicating device for reproducing the quotient in visible form.

It has been found expedient to dilute the sample liquid before the measurement in order to avoid crystallization, and this can be accomplished automatically.

The measurement of the angle of rotation α and of the refractive index n can be effected either simultaneously by parallel flow of the sample to be measured or successively by connecting the measuring points in series.

In the automatic formation of the quotient $\alpha/(n-n_0)$ it is advantageous to measure the temperature on the material constantly and to automatically correct for the influence of the temperature on the quantities α , n and n_0 .

Referring now to FIG. 2, magnetic valves 1, 2, 3, 4, and 5 are arranged respectively in five feedlines for different samples. These valves are actuated selectively according to a preset program which is controlled, for example, by a process computer. The various feedlines for the samples open into a common conduit 6, having a pump 7 therein, and connected to a mixing device 11. A pure solvent, such as water, is also supplied to the mixing device 11 through another conduit 8 having a pump 9 therein. The pumps 7 and 9 are driven by a common motor 10 so that the same amount of solvent is always added to a given amount of sample liquid. The mixing device 11 is driven by the motor 12 and may be designed as a circulating pump.

From the device 11 suitable conduits 35, 13 at the same time carry some of the diluted sample liquid into a measuring cell of a polarimeter 14 and the rest into the measuring cell of a refractometer 15. The temperature of the conduits 35, 13 and of said measuring cells is controlled, so that there is no temperature drop between the measuring points.

The polarimeter 14 is so constructed that it shows the measured value of the angle of rotation α in digital form at 16. At the same time it produces the measured value in its analog form as an electric voltage tapped by a precision potentiometer 17.

The refractometer 15 measures the deviation of the refractive index n of the sample liquid from the refractive index n_0 of pure water. It produces the measured value $n-n_0$ in its analog form as an electric voltage tapped by a precision potentiometer 18.

The electric voltages produced by the measuring instruments 14, 15 are processed in a calculating device 19 which calculates the quotient $\alpha/Cn-n_0$ electrically. The device 19 contains resistances 20, 21 connecting one end of the output network (viz resistor 26) in series with one end of the potentiometer 17; device 19 also includes a resistance 22 connecting the other end of the output network (viz resistor 29) in series with one end of the potentiometer 18. The tap of the potentiometer 17 is connected to resistance 23 while the tap of potentiometer 18 is connected to resistance 24. Resistances 23 and 24 are both connected to an amplifier 25 the amplification factor of which may be one, and the amplifier output is connected to the resistor 29 end of the output network. The midconnection of the output network is grounded, as are also the remaining ends of the potentiometers 17, 18. Values of the quotient $\alpha/(n-n_0)$ of less than 50 do not occur in practice, so that it is reasonable to suppress them for the recording. By means of the resistances 26, 27, 28, 29 this suppression is effected so that the output voltage at 30 is zero for the quotient 50 and only quotients of more than 50 produce an electrical voltage at the output 30.

By means of the resistances 26, 27, 28, 29 it is also possible to approximate the quotient $\alpha/(n-n_0)$ by means of a correction factor F to the value of the quotient (Pol/brix)·100 conventionally determined. The factor F may be adjusted by adjusting the resistances 26 and 29.

It is another object of the calculating device 19 to compensate for the temperature coefficient of the quotient. For this purpose a thermistor 31 is connected into the conduit leading the sample liquid from the device 11 to the measuring instruments 14, 15. This thermistor is connected in series to the resistances 20, 32 which are arranged within the calculating device 19. By means of thermistor 31 and resistances 20, 32 a temperature coefficient is produced which corrects automatically the influence of the temperature of the sample liquid on the quotient $\alpha/(n-n_0)$.

For automatic regulation or control of the sugar-manufacturing process an appropriate process control computer (not shown) would have its input connected to the output 30 of the calculating unit 19.

In the embodiment illustrated, an analog-digital converter 33 is connected between the calculating unit 19 and an indicating device 34 which shows in digital form the quotient

$$\frac{\alpha}{n-n_0} \text{ or } \frac{\alpha}{n-n} \cdot F$$

calculated in the unit 19. Alternatively, the analog digital converter 33 could be omitted and the indicating device 34 could be replaced by an indicating device adapted to show the quotient in analog form.

What is claimed is:

1. A method for measuring the proportion of sucrose in the total of dissolved substances of intermediate products in sugar manufacture comprising measuring refractometrically the refractive index n of a sample solution of an intermediate product which is to be measured, measuring polarimetrically the angle of rotation α of said sample, and combining these measurements and the known refractive index n_0 of pure water in the quotient $\alpha/(n-n_0)$.

2. The method of claim 1 in which the difference between the refractive indices n and n_0 is directly measured refractometrically and in which this difference and the polarimetrically measured angle of rotation α are in the form of electric quantities, and in which the respective electric quantities are combined electrically to produce an electric signal representing the quotient $\alpha/(n-n_0)$.

3. The method of claim 2 in which the electric signal representing said quotient is produced in analog and in digital form, whereby said analog form is available for the application to control the operation of a process control computer and said digital form is available for operating an indicating device for reproducing said quotient in visible form.

4. The method of claim 1 in which said sample is diluted before measurement.

5. The method of claim 1 in which said quotient $\alpha/(n-n_0)$ is produced in the form of an electric quantity and in which said quotient is electrically multiplied by a factor F in order to relate said quotient to the conventional purity quotient (Pol/Brix)·100.

6. The method of claim 1 in which the difference between the refractive indices n and n_0 is directly measured refractometrically and in which this difference and the polarimetrically measured angle of rotation α of the sample solution are reproduced in the form of electric quantities, and in which the respective electric quantities electrically produce an electric signal representing the quotient $\alpha/(n-n_0)$, further comprising measuring the temperature of the sample solution, reproducing electrically a temperature factor, and multiplying said quotient by said factor electrically in order to correct the influence of the temperature of the sample solution on said quotient.

7. The method of claim 2, in which the electrical signal representing said quotient is produced in analog form.

8. The method of claim 2, in which the electrical signal representing said quotient is produced in digital form.

9. The method of claim 1, in which the difference between the refractive indices n and n_0 is measured refractometrically, and in which said combining step combines said last-mentioned measurement and said polarimetric measurement.

10. The method of claim 9, in which a temperature measurement is made on the sample solution, and in which said combining step combines said temperature measurement with said quotient to correct the influence of the temperature of the sample solution on said quotient.

11. The method of claim 1, in which said measurements are made concurrently.

12. The method of claim 1, in which the sample solution is continuously flowing and in which said measurements are continuously made on separate like divisions of the sample flow, the first-mentioned measurement being a refractometric measurement of the difference between the refractive indices n and n_0 in one of said flow divisions, the second-mentioned measurement being made in another of said flow divisions.

13. The method of claim 1, in which said measurements are made at the same temperature.

14. Apparatus for measuring the proportion of sucrose in the total of dissolved substances in liquid intermediate products in sugar manufacture, comprising a mixing device, first conduit means connected to said device for supplying to said device a sample flow of one such intermediate product having an unknown sucrose content, second conduit means

connected to said device for supplying a flow of pure water to said device, refractometric means including a measuring cell connected to the output of said device and accommodating mixed liquid discharge thereby, polarimetric means including a measuring cell connected to the output of said device and accommodating mixed liquid discharged thereby, said refractometric means producing an electrical signal output proportional to the difference between the refractive index n of the sample liquid and the refractive index n_0 of pure water, said polarimetric means producing an electrical signal output proportional to the angle of rotation α of the sample liquid, and quotient-calculating means electrically responsive to said signal outputs and producing an output indicative of the quotient $\alpha/(n-n_0)$.

15. Apparatus according to claim 14, in which said cells have separate and like connections to the output of said mixing device, whereby they are simultaneously subjected to like but divided flows of diluted sample liquid.

16. Apparatus according to claim 15, in which heat-responsive means producing an electrical signal output is positioned at the output of said mixing device, said heat-responsive electrical signal output being connected in temperature-correcting relation to said quotient-calculating means.

17. Apparatus according to claim 14, in which said conduits each include a pump, said pumps having a common drive.

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