

DETERMINATION OF SUCROSE IN MIXED JUICE AND FINAL MOLASSES BY THE JACKSON AND GILLIS (METHOD IV)

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Abstract

Samples of mixed juice and final molasses were analysed by the Jackson and Gillis (Method IV) and the results were compared with the gas chromatography results, obtained by the Sugar Industry Central Board and the Sugar Milling Research Institute. Good correlation coefficients of 0,992 and 0,972 were found for mixed juice and final molasses respectively, although the statistical results exhibited a bias. The technique is relatively simple and apart from a temperature controlled water-bath for inverting the solutions, all the necessary apparatus and reagents are already in use in factory laboratories.

Historical Note

Sugar factory products generally contain three main optically active sugars, viz fructose, which rotates the plane of polarisation of polarised light to the left and glucose and sucrose which both rotate this plane to the right. The direct polarisation of a product will therefore be the resultant polarisation of these three sugars. The principle of double polarisation was standardised by Clerget in 1846 (Meade¹) in which this direct polarisation was supplemented by a second polarisation after inversion of the sucrose. Since the rotation of the non-sucrose remains constant, the change in polarisation would be due to the sucrose in the product. Although the theory of the method is simple it proved to be troublesome, especially with low grade products such as cane molasses in which there are appreciable quantities of amino compounds. Many modifications of the method were undertaken, one of which was the Jackson and Gillis (Method IV) (J & G) where neutral salts were added to offset the presence of the hydrochloric acid. This method was adopted world wide, but was eventually replaced by the chemical method of Eynon and Lane. The results by this method, which also involves acid inversion, over-estimate sucrose in final molasses as obtained by gas chromatography (GC) by about 2,5% (Schäffler & Loker²). The Jackson and Gillis (Method IV) has long since been rejected by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) as not being sufficiently accurate, but with the advent of more sophisticated and accurate methods such as GC, the technique was reinvestigated in order to give mill laboratory staff another tool which could be used to determine sucrose more effectively.

Introduction

The Affiliated mills send composite samples of their final molasses to the Sugar Milling Research Institute (SMRI) each month for analysis which includes the determination of sucrose by the titration method of Eynon and Lane after inversion with hydrochloric acid. A project was undertaken to determine whether the agreement of results obtained using the Jackson and Gillis (Method IV) would be closer to those obtained by gas chromatography. The project was also extended to include the analysis of mixed juice where GC sucrose is determined by the Sugar Industry Central Board (SICB). Since GC analysis of samples from the Affiliated

mills is not undertaken routinely, samples of South African origin were used in order to lighten the load of the Chemical Division of the SMRI. The authors feel that the composition of juice and molasses from the Affiliated mills does not differ greatly from that of the juice and molasses produced in South Africa.

Method

Samples of final molasses which are sent to the SMRI on a weekly basis for a full analysis were selected at random and analysed, as were mixed juice samples obtained from the SICB. Altogether 48 samples of molasses and 29 samples of juice were analysed during the 1987/88 season. During the first few weeks of the 1988/89 season further samples were analysed from three South African mills viz Darnall (DL), Illovo (IL) and Union Co-op (UC) and the results were compared with their respective GC figures. The analytical method is set out in detail in the Appendix.

Results and Discussion

Although t-tests done last season showed the comparisons to have highly significant differences, the correlation coefficient for mixed juice was 0,988 (mean GC sucrose = 9,66% and J & G sucrose = 9,49%) and the coefficient for final molasses was 0,979 (mean sucrose = 28,55% and 28,16% for GC and J & G respectively).

This season samples were selected from DL, IL and UC as being representative of mills in the north, south and interior regions of the South African sugar belt and all the results were used for statistical analysis. The t-tests were again highly significant but the new correlation coefficients

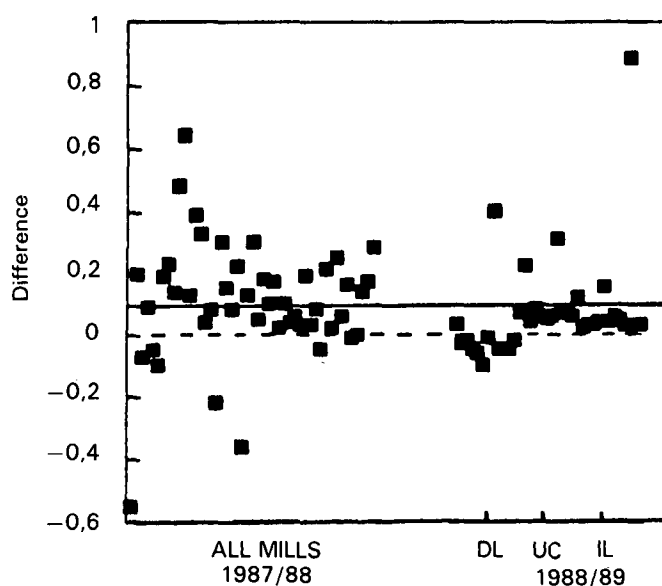


FIGURE 1 Discrepancies between sucrose analyses obtained with the GC and J & G methods applied to mixed juice from various factories and seasons.

determined, viz 0,992 and 0,972 for juice and molasses respectively, were good. The mixed juice regression formula is $Y = 0,297 + 0,976 \cdot X$ with a standard error estimate of 0,16 while the final molasses regression formula is $Y = 1,1 + 0,946 \cdot X$ with a standard error estimate of 0,35.

The mean difference between sucrose % mixed juice determined by GC and J & G is 0,092 which is 0,9% relative to mixed juice. Figure 1 shows the differences between GC and J & G on all of the mixed juice samples analysed during both seasons with the mean difference shown as a solid line. The scatter from mill to mill is evident.

Figure 2 shows the relationship between the J & G results for sucrose and the respective pol values. The pattern is slightly different to Figure 1 and the mean difference (again shown as a solid line) is less at 0,065.

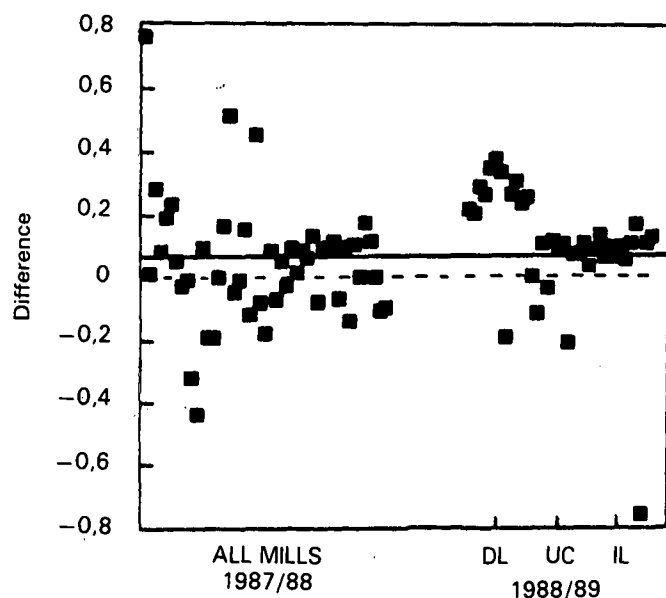


FIGURE 2 Discrepancies between sucrose analyses obtained with the J & G method and the pol of mixed juice from various factories and seasons.

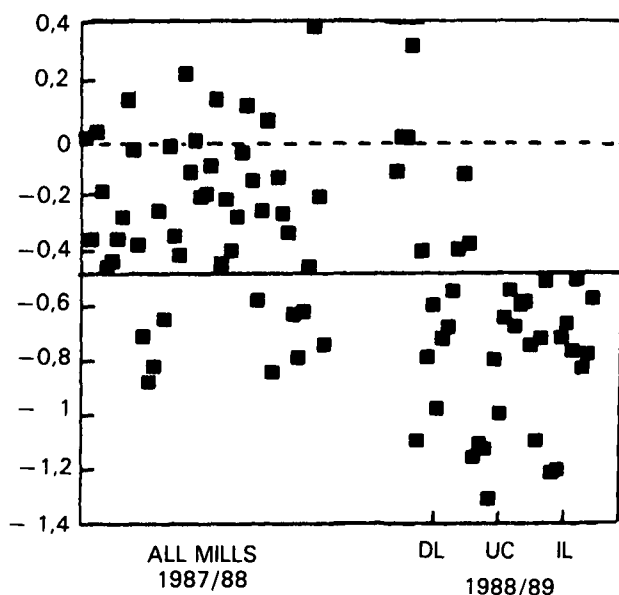


FIGURE 3 Discrepancies between sucrose analyses obtained with the GC and J & G methods applied to final molasses from various factories and seasons.

Pol/sucrose ratios calculated using the J & G results are higher than those calculated by GC (mean pol to sucrose ratios using J & G and GC results are 0,9937 and 0,9836 respectively). The mean J & G to GC ratio is 0,9906. The values of GC sucrose - J & G sucrose in the final molasses are exhibited in Figure 3 with the mean difference (-0,478) as the solid line. A strong bias from mill to mill as well as from season to season is evident.

The mean differences for the 1987/88 and 1988/89 seasons are -0,287 (1,0% relative to molasses) and -0,733 (2,5% relative to molasses) respectively. The mean difference over both seasons (-0,478) is 1,7% relative to final molasses and is thus lower than the 2,5% over-estimation of sucrose found by the chemical method.

Conclusions

The statistical analysis of the results obtained in this exercise indicate the following:

- The t-tests show significant differences in sucrose determined by the Jackson and Gillis (Method IV) when compared with those found by gas chromatography for both mixed juice and final molasses although regression coefficients are good
- The molasses results show a bias from season to season
- The average discrepancy between the two methods is 0,478 units, i.e. 1,7% relative for molasses and 0,092 (0,9% relative) for mixed juice
- Although the mean relative difference between J & G and GC in final molasses (1,7%) over both seasons is lower than the 2,5% over-estimate found using the chemical method, it was as high as 2,6% for the three mills tested during the 1988/89 season
- From the regression analysis there appears to be quite good agreement between the J & G and GC methods for the determination of sucrose in mixed juice although the J & G method on average under-estimates the sucrose by 0,9% relative
- The double polarisation method is apparently slightly better than the chemical method of Eynon and Lane for determining sucrose in final molasses, and could therefore be used by the Affiliated mills to reach a more accurate measurement of sucrose in mixed juice and final molasses.

The double polarisation technique is relatively simple to develop and apart from the necessary "A" grade glassware and a temperature controlled water-bath for inversion of the solutions, all apparatus and reagents are already used in sugar mill laboratories.

REFERENCES

1. Meade, GP (1963). *Cane Sugar Handbook*, Ninth Ed., John Wiley & Sons, Inc., New York. Chapter 22.
2. Schäffler, KJ and Loker, C (1974). Quantitative Gas Chromatography of Carbohydrates in Cane Molasses. *Proc Int Soc Sug Cane Technol*, XV 1380-1387.

APPENDIX

Determination of Sucrose in Mixed Juice and Final Molasses by the Jackson and Gillis (Method IV)

Reagents: Basic lead acetate powder
Sodium chloride solution, 231,5 g/litre
Potassium oxalate
Hydrochloric acid, 1,1029 SG
Acetic acid, 1 M

- Apparatus:**
- 500 cm³ Erlenmeyer flask and stopper
 - Pol dish
 - 250 cm³ volumetric flask and stopper
 - 2 × 250 cm³ tall form beakers
 - 2 × stemless funnels, 100 mm dia.
 - 2 × watch glasses, 100 mm dia.
 - Filter paper, Whatman No. 91 or equivalent
 - Filter paper, Whatman No. 6 or equivalent
 - 2 × 100 cm³ "A" grade sugar pol flasks
 - Saccharimeter or polarimeter with 200 mm water jacketed pol tube
 - 50 cm³ pipette
 - 2 × 10 cm³ pipettes

A: MIXED JUICE

1. Measure the brix of the mixed juice and record the result.
2. Pour 150 cm³ mixed juice into the Erlenmeyer flask. Add 1,5 g of dry basic lead acetate powder, stopper and shake well. Leave to stand (in the constant temperature room if possible) for a few minutes.
3. Filter through the Whatman No. 91 filter paper, rejecting first runnings, until the drops are clear. Take care to eliminate evaporation by covering the funnel with the watch glass.
4. Carefully pipette 50 cm³ of the filtrate into each of the two sugar pol flasks. Mark them "D" and "I".
5. To the first flask marked "D", add 10 cm³ sodium chloride solution. If the solution becomes cloudy after the addition of the sodium chloride, clear with a drop or two of acetic acid. Set aside.
6. To the flask marked "I" add 20 cm³ distilled water. Heat in a water bath to 65°C and immediately add 10 cm³ hydrochloric acid. Mix by rotating the flask in the water bath and set aside for at least 30 minutes.
7. Make both flasks almost to the mark with distilled water and place them in a water bath at 20°C for half an hour.
8. At the end of the half hour, carefully make both flasks exactly to the mark, dry the necks with filter paper and shake thoroughly.
9. Read both solutions in the water jacketed pol tube at 20°C. Multiply these readings by 2 and record them as "Dr" and "Ir".
10. Using Schmidt's table convert the "Dr" and "Ir" readings to "Pd" and "Pi" respectively, and calculate the % sucrose from the formula in section C below.

B: FINAL MOLLASSES

1. Weigh 32,5 g molasses into a pol dish, dissolve in some distilled water and transfer quantitatively to the 250 cm³ volumetric flask, make to the mark, stopper and shake well.

2. Weigh 15 g basic lead acetate powder into the Erlenmeyer flask. Pour the contents of the volumetric flask into the Erlenmeyer flask containing the basic lead acetate powder, stopper, shake well and leave to stand in the constant temperature room (if available) for about 5 minutes.
3. Filter the contents of the flask through the Whatman No. 91 filter paper discarding first runnings and taking care to eliminate evaporation by covering the funnel with the watch glass.
4. When all of the solution has filtered through the filter paper, dissolve 5 g of solid potassium oxalate in the filtrate and refilter through the Whatman No. 6 filter paper, again discarding the first runnings and covering with a watch glass as before.
5. Proceed as for mixed juice, steps 4 to 8 inclusive.
6. Read both solutions in the water jacketed pol tube at 20°C. The saccharimeter readings multiplied by 4 in each case will give "Pd" and "Pi".
7. Since the accuracy of the determination of sucrose in final molasses by this method is not very great, no significant error is made if a constant value of 132,0 for the divisor is used to calculate the sucrose % using the formula in section C below.

C: CALCULATION

The calculation is done as follows:

$$\text{Sucrose} = \frac{100(\text{Pd} - \text{Pi})}{132,63 + 0,0794(m - 13) - 0,5(t - 20)}$$

where t = the temperature of the solution at the time of polarisation and
 m = the total weight in grams of the dry substance in 50 cm³ of the undiluted juice.

If the measurements are carried out at 20°C, the divisor in the above formula can be obtained from Table 1.

Table 1

Table giving the divisor, corrected for the brix of the solution, of the formula to be used in the calculation of sucrose by the Jackson and Gillis (Method IV)

Brix	Divisor	Brix	Divisor	Brix	Divisor
0	131,60	9	131,97	18	132,36
1	131,64	10	132,01	19	132,41
2	131,68	11	132,05	20	132,46
3	131,72	12	132,10	21	132,50
4	131,76	13	132,14	22	132,55
5	131,80	14	132,18	23	132,60
6	131,84	15	132,23	24	132,64
7	131,88	16	132,27	25	132,69
8	131,92	17	132,32	26	132,74