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QUALITY CONTROL REQUIREMENTS OF THE SUGAR INDUSTRY 1/

by

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id. 74-

SUMMARY

Quality of raw material, product and in-process materials are identified as separate areas of specialisation.

Purchase of raw material introduces direct financial factors. Inbuilt incentive systems are considered especially for cane supply.

Quality of product white sugar varies according to local needs.

Thirteen quality requirements for raw sugar are listed.

Process quality control involves numerous decisions relating to precision required and the cost of higher precision related to the value of data.

Sampling errors need specific evaluation.

Undetermined loss if real is costly, but may be the net result of imprecision in individual loss assessment, quality of raw material and quality of product. The law of propogation of errors needs to be applied and its implication understood.

A. Introduction

Quality control requirements may be considered under three areas viz:-

1. Quality of raw material
2. Quality of product
3. Quality of in-process materials

B. Raw Material

These may be considered under three headings:-

1. Sugar beet evaluation
2. Sugar cane evaluation and cane payment
3. Raw sugar evaluation as a raw material for a refinery

C. Sugar Beet Evaluation

The evaluation of sugar cane and its related payment system will be discussed in rather more detial than the corresponding relationships in the sugar beet industry much of which will cover the general principles which are involved in these procedures.

On the other hand there are certain specific differences in the nature of the raw material which makes it desirable to give separate mention to some of the problems specifically related to sugar beet.

Sugar beet is a root vegetable rather than a stalk of grass. For this reason harvesting is an entirely different type of operation. Climatic conditions are also vastly different and influence the manner of collection, transport and storage.

The primary object however is to determine the amount of sucrose in the beet and what benefit from a good quality product or [illegible] for poor quality product.

For this purpose the whole beet must be sampled rather than taking an empirically related extraction juice quality.

Sampling of sugar beet itself may or may not be a less difficult operation than sampling sugar cane, this will be largely a matter of opinion.

Extraneous matter with sugar beet will be largely soil and stones with possibly some residual stalk or leaves and such residues of the root system as may be retained. Extraneous matter of course will not produce sugar but it is not easy to make an accurate evaluation. However if total extraneous matter is kept to less than say 2% of the total weight of raw material then a precision of ±10% in the evaluation of this extraneous matter will enable the over-all precision to be maintained to within ±0.2% which should generally be an acceptable figure at this point.

Sampling the whole beet has been the subject of special study in this area and various techniques are possible. It does lend itself reasonably well to statistical study of data in order to be able to specify the precision of the sampling technique.

The beets must be sub-sampled and the juice extracted in order to analyse its quality and proportion in the whole beet. These techniques have been systemetized so that a large number of samples may be processed in a given time and automatic polarimetry is now a common technique for measurement. Associated mechanical recording of data may be included.

Because of the lower "fibre" content of sugar beet the juice analysis is probably more representative of the whole plant than is the case with sugar cane. Furthermore it is easier to obtain a representative sample from a whole plant.

Polarisation is, however, used to represent the sucrose in the beet. This is also probably much more satisfactory than is the case with cane juice since hexoses do not appear in beet juices unless determination has resulted in hydrolysis of sucrose. There may be small amounts of other optically active materials but of much less significance than is the case with cane juices.

Climatic conditions such as heavy frosts and maturing requirements frequently require beet to be harvested faster than it can be processed resulting in stockpiling at the factory. The cold climate does enable this to be done with minimum of determination, nevertheless an assessment of the deterioration is important and should not be overlooked when assessing the quality of the beets themselves.

D. Sugar Cane Evaluation

Much cane is purchased simply on a gross tonnage basis especially in developing countries. Its only advantage is simplicity. By allowing no direct payment for sugar in the cane there is no incentive for the farmer to give attention to growing better quality cane or to minimise deterioration between time of harvesting and time of processing. This is aggravated when it is the responsibility of the farmer to deliver the cane to the factory.

It also does not discourage the supply of dirty or poorly topped cane. In developing countries where field wages are low and holdings are small the cane is very often well prepared by way of good topping and complete removal of trash, it is not unknown for the cleaned stalks to be wrapped neatly in bundles. This cleanliness disappears as size of operations increase and when mechanical harvesting is introduced the cleanliness of the cane is largely related to the type of harvesting equipment. Simple grab or rake harvesting will gather everything indiscriminately and it is necessary to spend large sums of money on cane cleaning plant at the factory.

At the other extreme, modern chopper type harvestors which cut the cane into short lengths in the field are fairly effective in removing trash and topping is not unreasonable. On the other hand the chopped sticks are very susceptible to deterioration and should be processed within not more than 30 hours of harvesting.

Quality evaluation of cane does confer many benefits and is the obviously desired approach but is difficult to implement especially with precision. There are two general approaches to this problem. One is to estimate the quality of cane from an empirical relationship between the juice expressed at the first roller of the milling train and the total sugar in cane. The other is to attempt to sample the whole cane and carry out an analysis. In between are proposals involving such a procedure as expressing juice from a whole cane sample under controlled pressure conditions of a hydraulic press.

The procedure which has held favour for the longest period of time is the one adopted by the sugar industry in Australia and known as the C.C.S. Formula (Commercial Cane Sugar) this was developed around the turn of the century from a study of empirical relationships between the purity of first expressed juice and the recoverable sugar in the cane. The fibre content of the cane also needs to be known.

The formula depends on the assumption that for every unit of soluble impurities in the cane one half of a unit of sucrose is not recovered in the process.

$$\text{Hence C.C.S.} = \text{sucrose in cane} - \frac{\text{impurities per cent cane}}{2}$$

In the normal application of the formula the following assumptions are made:-

1. Brix = Total soluble solids (dry substance)
2. Sucrose = Pol
3. Impurities = Brix - Pol
4. Brix per cent cane:-

$$= \text{Brix per cent first expressed juice} \times \frac{100-(F+3)}{100}$$

5. Pol per cent cane:-

$$= \text{Pol per cent first expressed juice} \times \frac{100-(F+5)}{100}$$

Hence

$$\text{C.C.S.} = \text{Pol in cane} - \tfrac{1}{2}(\text{Brix in Cane} - \text{Pol in Cane})$$

$$= \frac{3P}{2}\left(1-\frac{F+5}{100}\right) - \frac{B}{2}\left(1-\frac{F+3}{100}\right)$$

where P = Pol per cent first expressed juice

B = Brix per cent first expressed juice

F = Fibre per cent cane

A more simple relationship known as the Jave Ratio is the percentage ratio of the pol per cent cane to the pol per cent first expressed juice:

$$\text{Hence} \quad \text{Pol in Cane} = \text{Pol per cent first expressed juice} \times \frac{\text{Java Ratio}}{100}$$

This does not make any adjustment for variations in the fibre per cent cane and loses much of its usefulness when these variations become significant.

The fact that the C.C.S. formula has certain imperfections is well known but it is considered to be better to live with these imperfections than to discard the formula.

The alternative of whole cane analysis has probably been developed to a more advanced degree in South Africa than in any other country. The technique involves taking a sample right across the chute after the shredder, passing this through a sample shredder, sub-sampling and performing an ambient temperature water extraction in a high speed blender. The temperature does riseto about 65°C during this treatment but this is considered to be acceptable.

It is known that the procedure is costly, it is not yet known whether the cost is justified by the value of the results.

One of the better intermediate procedures is that recommended by Hugot as follows:-

1. Use of a core sampler of the Hawaiian or American or the French JAMPO.
2. A sampling probe - 150 or 200 mm diameter tube which can revolve at 450 or 600 rpm while moving forward, with the mouth fitted with a saw all around its edge.
3. The sample disintegrated in a cutter grinder of prescribed make.

4. A quantity of 1kg weighed and transferred to a press pot with a large number of holes with application of pressure equal to 400 bar for 90 seconds.
5. Juice analysed for refractometric brix and pol by an electronic automatic polarimeter. Presscake is removed and weighed and the fibre determined by a correlation scale or formula.

Hugot further recommends adopting ideal efficiency corresponding to Reduced Mill Extraction of 97.5; no loss in filter cake, no undetermined losses and the final molasses exhausted to about 29 apparent purity.

The formula recommended by him for direct analysis is:-

$$R_s = K(1 - 1.45f)(S - 0.3B)$$

The coefficient 1.45 applies to the 400 bar press and should be suitably modified for other pressures, being higher for lower pressures and vice versa.

An interium report prepared by the Committee on the Payment of Cane Price on a Quality Basis set up by the 1971 Congress of the I.S.S.C.T. was submitted at the 1974 Congress which reports on what is being done in many of the sugar cane growing countries. The study is still proceeding.

Meantime the Australian C.C.S. formula probably provides the best empirical assessment with the advantage of a built in incentive for good quality cane by virtue of the impurity adjustments.

To implement the system effectively requires every delivery of cane to be kept distinct and identifiable, right to the first crushing unit of the milling train.

Cane piling, the use of cleaners and other convenient devices to improve the quality of cane or to reduce handling costs are incompatible with the system.

Regulation 51 was amended in 1957 to recognise the C.C.S. figure derived from the juice weight control for the week as the official average for cane payment purposes. The average of all the individual C.C.S. determinations for the week is compared with the average according to the juice weight system. The ratio of these two constitute a "Correlation factor" which is applied to all individual determinations for the week. Recently there have been "second thoughts" on the precision of juice weigher results and more emphasis is given to actual sugar recovered.

The following refinements in the International methods have been introduced:-

1. In solubles - "dirt" in mixed juice determined and treated as fibre.
2. Mixed juice filtered or centrifuged before brix is determined by gravity spindle.
3. Purity of last expressed juice is not a satisfactory substitute for the purity of residual juice in bagasse for the assessment of loss in bagasse.

An incentive payment system is only partially effective by application of a formula such as that of C.C.S. In fact the built-in incentive is not sufficiently conspicuous to be observed by the farmer and he must be instructed in its effect.

The other part of incentive payment is in the formula used to calculate payment itself. Here again the Australian system has substantial advantages especially in association with the C.C.S. assessment.

The basic price of sugar cane of average C.C.S. is determined in accordance with the following formula:-

$$V = \$0.009\ P(a-4) + \$0.333$$

where V = the price of cane in dollars per ton

P = the price of sugar in dollars per ton

a = the actual mill average C.C.S.

The price of cane in relation to C.C.S. is then calculated by a formula of the following type:-

$$v = V(C.C.S.-k)$$

This provides a very distinct price incentive for cane of high quality and dis-incentive for cane of low quality. The effect of this is much more conspicuous to the farmer and is the really influential part of the system in encouraging the supply of good quality cane to a sugar factory.

E. Sugar Product Quality

Refined sugar which appears on the domestic market can be as pure as any "Analytical Reagent" grade of chemical. The highest purity is not necessarily required for domestic use, but is desired for use in the manufacture of beverages, preserves and related food products. It must be fre of oolour, perfectly sweet not result in any precipitation from beverages.

To a certain extent sweetness and colour are inter-related. The colour components associated with sucrose are very bitter in taste and as most of the colour is on the surface of crystals there can be differences in the taste of crystals for this reason. Different sized crystals may also differ in apparent taste because small crystals dissolve faster than large ones.

For quality control purposes colour is measured in solution with a spectrophotometer and a wave length of 420 nm has been accepted as the standard for measurement.

Purity as measured by polarisation and moisture determination is probably next in importance to colour determination.

Hexose concentration (as reducing sugar) and ash are also measured. The concentrations are very low and accurate analysis is difficult. Colourimetric "trace" test techniques are often necessary for hexose analysis of the best quality sugars, whilst conductivity techniques become necessary for ash analysis.

The size of crystal also has importance to customers. Just what size range is desired varies from one country to another and represents a variation in customer preferences. Large crystals generally have a better keeping quality than small ones by virtue of their lower specific surface.

In many developing countries a lower quality "white" sugar is commonly marketed and is adequate for most purposes. Sometimes the crystals may be markedly grey or yellowish in colour. These sugars are not suitable for beverage or preserve manufacture and their keeping quality is not of such high standard. Were it not for the fact that most of these sugars are of large crystal size the poor keeping quality would be much more conspicuous.

Uniformity of crystal size is maintained more often by sieving out of smaller fractions than effective sugar boiling control.

The analytical requirements are still those of colour, polarisation, moisture, hexose (as reducing sugar) ash and sieve analysis.

F. Raw Sugar Quality

Raw sugar is the commodity which dominates the world sugar market, being produced by factories in tropical countries for refineries usually situated in cool to cold temperate zones. In recent years refineries have begun to appear in tropical areas many of which are completely independent of sugar milling factories.

It is undoubtedly a fact that the criterion of greatest concern to both buyer and seller is the price. This has varied so much between the lows and the highs that by comparison variations in quality are largely marginal.

Three aspects are relevant in these considerations:-

1. The actual value of raw sugar as an independent commodity.
2. The operating costs of a refinery as a separate financial organization.
3. The marginalccost of the refining process when carried out as an on-site extension of raw sugar production.

Nevertheless it is possible to identify thirteen distinct chemical and physical criteria which are important in the buying and selling of raw sugar. These are:-

1. Polarisation
2. Moisture content
3. Total impurities content
4. Concentration of specific impurities
5. Concentration of specific impurity components
6. Colour value of whole crystal
7. Colour distribution within crystal
8. Concentration colour inclusions
9. Average crystal size
10. Uniformity of crystal size
11. Crystal shape
12. Mono-crystalline character-conglomerate effect
13. Filterability

The first criterion listed - that of polarization - probably carries as much weight as all other twelve put together. Customs duties imposed by importing countries are usually based on polarization values. It is well known that polarization is only a physical measurement and is influenced by non-sucrose constituents. The influence is probalby greater in the case of sugars originating from cane than for sugars of corresponding quality originating from beet, owing to the virtual absence of hexose sugars in beet juices.

Means have been developed, and are being used, for the adjustment of pol values of raw sugars to enable them to meet all other twelve quality criteria, especially that of filterability, and still come below the dutiable limit of polarisation. Such "doctoring" of sugar can usually be identified from a study of colour/impurity ratios and other analytical criteria. The hexose/ash ratio may also indicate abnormalities introduced for this purpose.

G. Process Quality Control

Quality control within a process between the limits of raw material supply and product delivery are important chiefly from the point of view of the materials balance sheet. Materials leaving the process such as pulp or fibre, mud, molasses and undertermined loss all need careful evaluation. This evaluation requires a knowledge of quantity as well as quality.

Bagasse from a cane sugar mill represents a much larger bulk of material than the residual pulp from a beet sugar factory. Bagasse is difficult enough to sample and analyse correctly but is even more difficult to weigh. It is common practice to evaluate the quantity from the fibre analysis of cane supplied. This can be only as precise as the fibre analysis itself which is often suspect in terms of precision. The bagasse is not worth weighing for its own sake and because the quantity of sugar being lost with the bagasse is relatively small the economics of precision weight measurement are usually considered to be very doubtful.

This makes it difficult to make a good assessment of milling behaviour especially in relation to possibly competitive diffusion operations.

Mud discarded from the filter operations of a factory are usually evaluated satisfactorily from the point of view of both weight and analysis.

Molasses has its own particular difficulties of assessment. It is commonly weighed - not easily, but may be measured volumetrically in tanks. The latter procedure also has inherent difficulties such as the entrainment of air bubbles which deflate the density.

Still more unsatisfactory is the commonly accepted procedure of analysis which involves the measurement of polarization rather than sucrose. In the case of beet molasses the error is probably marginal because of the virtual absence of hexose sugars. On the other hand cane molasses has a substantial difference between pol and sucrose, the difference increasing as the concentration of hexose increases and further affected by the ratio of glucose to fructose within the hexose group of sugars. A high fructose ratio still further depresses the polarisation value.

Sucrose analysis is difficult and accurate analysis is very difficult. Many laboratories in developing countries are inadequately equipped and inexperienced for sucrose analysis. This makes it very difficult indeed to make an effective assessment of operating criteria in the absence of such information.

The exhaustibility of final molasses is also related to the hexose/ash ratio and these groups of components should be the subject of analysis for effective quality control.

Within the process individual unit operations have special analytical requirements for their effective control. Extraction at diffusers or milling trains require the analysis of pulp or bagasse for residual sugar. Residual non-sugars and moisture analysis are also important in the case of bagasse coming from a milling train in order to evaluate performance effectively.

Within a milling train itself a knowledge of the moisture and pol of bagasse leaving each mill as well as the pol and brix of expressed juices are desirable for individual mill control.

The quality control of juice clarification involves a knowledge of pH and of turbidity of clarified juice. Purity analysis is usually performed but is of little value for evaluation of the quality of the clarification process because of the small differences in these figures.

The brix of clarified juice and of syrup leaving the evaporators is necessary to evaluate the performance of the evaporators.

Sugar boiling operations are usually controlled from purity and brix values of massecuites and syrups. This may be moderately satisfactory for white sugar boiling in a refinery or for sugar boiling in a beet factory, but it is less than satisfactory for controlling the boiling of syrups in a raw cane sugar factory or in the recovery end of a sugar refinery operating on raws which originated from sugar cane.

Here again the difficulties of actual sucrose analysis preclude its use for operating purposes and rule-of-thumb adjustments made to accommodate the errors of pol and brix values accentuated as the purity gets lower with each successive stage of boiling.

H. Sampling Errors

Sampling of products at any stage of sugar manufacture is difficult. Where buying and selling is involved as in the case of beets or cane on the one hand or product sugar on the other, well defined procedures for sampling have usually been set down.

Effective statistical evaluation of the precision of the various sampling procedures is just as important as the evaluation of the precision of analytical procedures.

I. Undertermined Loss

When preparing the balance sheet of materials entering and materials leaving the factory it is always difficult to close the balance precisely. The proximity of closure is the net result of all errors accumulated through the various procedures. More often than not the net result shows an undetermined gain. In the case of cane sugar factories some of this loss is inherent in the practise of using pol rather than sucrose for calculating a materials balance. If sucrose is substituted for pol in the final molasses assessment the undetermined loss will be significantly less.

The precision of the undetermined loss is also influenced by the precision of the determination of the individual losses. It is worth while making a statistical analysis of the precision of these figures as this sometimes avoids spending time searching for a fictituous value of the undetermined loss and especially of fluctuations in this value the reasons for which may be hidden in the magnitude of the errors in each individual determination.

If the precision of each loss measurement is evaluated in terms of Standard Error, then the precision of the undetermined loss in terms of Standard Error may be evaluated according to the law of propogation of errors which is expressed as the square root of the sum of the squares of individual standard error values. If for any reason we so desire to use Standard deviation values instead of standard error this may be used correspondingly in the evaluation provided consistent values are employed.

Questions:

1. What criteria are important for quality control evaluation?
2. How best are these criteria evaluated?
3. Why have we selected the specified criteria?

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