RAW CANE SUGAR QUALITY IN RELATION TO REFINING REQUIREMENTS

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I. INTRODUCTION

There is obviously a relationship between the type of raw sugar that is taken into a cane sugar refinery and the difficulties that are encountered in turning it into good quality white sugar. The factors affecting this relationship have been studied for many years, and efforts have been made by the raw sugar producers to supply to the refiners sugars with increasingly improved refining characteristics, even to the extent of sugar tailored to individual refining companies' separate needs. Raw cane sugar users, for their part, are beginning to build into their contracts of purchase specifications for the sugar delivered, with cash penalties on the producer if the sugar falls outside these agreed limits. Although it would seem logical to offer premiums for better than specification raw cane sugar, it is less likely for fiscal reasons and might even pose a threat to the very existence and livelihood of the refiners. Just as the specifications for the refiner's products become increasingly stringent, so he finds it necessary to apply more severe standards to his own raw material. In the sugar world, in this sense, there is a change from a seller's to a buyer's market. The days are disappearing, if not completely over, when the raw cane sugar refiner would accept anything—perhaps adding the proud boast that whatever it was he could refine it. The difficulties that this could lead to are illustrated by what used to happen in the British cane refineries in the years before the second world war. At that time, there was a seasonal pattern of white sugar production which was much more marked than today, the summer months, say from April to September, being much busier than the winter months. Unfortunately, summer was the time for receiving low quality Caribbean raws and the problems of decolorisation and efficient recovery work were immense. During the winter, when trade was slack, the main intake was higher quality sugar from the Southern hemisphere. Thus, so far as refining efficiency was concerned, the summer and winter intakes should preferably have been reversed. This pattern is not now so marked. For example, there is not such a great difference in demand for refined products between the two
seasons as there used to be, and there has been a tendency for raw sugar quality to become more standard. Nevertheless, the lot of the refiner can still sometimes become very difficult indeed, so that we should not be surprised if he seeks to make his job less onerous by applying some constraints on the quality of his raw material.

II. HISTORICAL

There is an extensive literature on raw sugar quality extending over many years. To make a complete review would be impracticable within the bounds of a review of acceptable length; however, it is desirable to make some selective references to the work which has been, and is being, done, if only to demonstrate the increasing interest in this subject and to show the changing emphasis on various factors over the years. There are various sources of published material: the normal publications of the technical press, the reports of professional associations or research institutes and the proceedings of various bodies connected with sugar production. It is difficult, if not impossible, to separate these into their various categories, and in the list which follows little attempt has been made to do so. However, the reports and proceedings of various selected bodies have been considered separately.

A. General

Possibly the earliest reference is an article in the International Sugar Journal (then The Sugar Cane) in 1898 in which Stein describes some early views on the relative values of raw cane sugar for refining purposes. Not surprisingly, there is no evidence of a follow-up to his pioneering until 1918 when Horne enumerated tests to evaluate raw sugar qualities from the refiner's point of view. In the same year, Browne published a long article on the bacterial deterioration of raw sugar in storage which, though published fifty-six years ago, is still comparatively applicable today. Peck in 1920 suggested means of improving the refining quality of raw sugar with particular reference to the filterability of affination syrup. In 1922, Well listed the ten main characteristics which he considered affected the refining quality of a raw cane sugar, namely polarisation, ash content, deterioration factor, grain, dark seed grain, colour, sulphates, filterability, caked and sticky raw sugar. Badollet and Paine in 1926 described the use of their dye test as a measure of filterability, and a little later Labayen described work being carried out in the Philippines to improve sugar quality for the American east coast refiners. Sengson in 1928 summarised the results of four years' study of the refining quality of Philippine raws. Rice in 1930 described tests designed to show the effect on refining quality of the impurity levels in six different raws, the chemical analysis of the sugars being related to such other factors as colloid measurement (dye number), colour, Elliott filterability and colour absorption by char. McAlle in

1931 described attempts being made in Hawaii to improve the quality of the sugar destined for Crockett refinery. In 1934, work was reported on Australian raws in which it was concluded that the pH level during clarification was the main factor in determining the filterability of the resulting sugar. About the same time, various papers were published from Australian sources listing techniques being used, including conductivity control of pan boiling, to improve the quality of raw sugar grain. Thorp in 1935 described work carried out by Mackay Sugar Manufacturers' Association in an attempt to improve the refining quality of raws produced in the Mackay district, and Hayes, at the same meeting, gave a paper on the refining quality of South African raws concluding that examination had failed to find any chemical constituent having a direct relationship to the filtration rate. Godchaux specified the three factors used in classifying raw sugar at the Reserve refinery in Louisiana, namely colour, solid impurities and filtration number.

There was a lull in publication between 1939 and 1945, an exception being an article by Webster in 1941 on the deterioration of raw sugar in storage. After the end of hostilities in 1945, an increasing interest was shown in the subject. Salinas suggested methods for improving the refining quality of Cuban raws, and Puertas, also in Cuba, collected and summarised data from the control sheets of nine factories in an attempt to list the factors affecting the filterability of raw sugar. In 1946, an anonymous article was published describing a process involving the use of a solvent to remove gums from cane juices and attempted a costing. It is interesting to note that such a process using carbon tetrachloride was proposed as long ago as 1928 by Gross at the 6th Annual Convention of the Philippine Raw Sugar Association. Stueber in 1951 listed factors which he considered important in avoiding difficulty in affination. These were grain size, freedom from conglomerates and freedom from foreign matter which could blind sugar and machine cloths and choke gutters. The Douwes-Dekkers reported the result of work carried out by the Sugar Manufacturers Research Institute in South Africa into the causes of deterioration of Natal raw and stressed the importance of good grain quality and of maintaining a satisfactory safety factor. In 1952, Kortschak discussed the effect of insoluble materials called colloids which could either be on the surface or included in the crystal. He concluded that poor filtration was due to the presence of particles too large to be properly termed colloidal, and blamed poor pan boiling methods and poor clarification for any excessive quantity of these materials in the raw sugar. Higginbotham in 1955 described a method of operating a raw sugar factory in a way designed to reduce the amount of fats, waxes and gums going through the process. Ishii in Japan tabulated the results of raw sugar analyses and studied the effect of insoluble particles of various sizes on filtration rates. Alexander in 1957 described filtration tests carried out on Natal Raws at the Sugar Milling Research Institute and concluded that phosphates do not affect filtration, while silica and wax do, while the effect of starch is doubtful. In an important article published in 1959, Davis made a lengthy review of work carried out by the Colonial Sugar Refining Co. on the
filterability of raw sugar. The filtration-impeding materials were considered to be the insolubles in sugar, solubles which become insoluble during refinery defecation (e.g. soluble phosphates), materials in sugar that affect viscosity at the filter station and insolubles formed in the refinery defecation process from materials added during refining. It is the refiner’s problem to persuade the raw sugar producer to reduce the amounts of the first three classes of impurity in his product. A study was made of the amount and effect of insolubles in raw sugar and also of starch, dextran and soluble phosphate, and a review was made of plant operations with suggestions that could improve filterability. In another paper published by the C.S.R. in 1961, Nicholson et al. discussed the relationship between filterability and filtration-impeding materials. Yamane et al., working in Japan, described work carried out on Natal, Australian, Taiwan and Cuban raws and gave graphs relating the filterability of raw and refined sugars to the amounts of gums, starch, silica, wax and phosphate present. In 1966 Kaga et al. published the results of work carried out on laboratory scale carbonation, from which they observed a close relationship between filterability of refined sugar and that of carbonation slurries and also between the starch content of refined sugars and the filterability of carbonation slurries produced from them. In the same year, the annual report of the Mauritius Sugar Industry Research Institute gave an account of the results obtained by the enzymatic removal of starch and demonstrated the effectiveness of the method for improving the filterability of raw sugar. Chang in 1968 investigated Taiwan raws and found that the only parameters which showed a significant correlation with filterability were the turbidity in solution and the silica content. Work published in the same year by the Tate and Lyle Research Centre confirmed that the main cause of the deterioration of freshly harvested cane was infection by Leuconostoc mesenteroides. Chen discussed the production of high quality raw sugar for refineries, the factors affecting the refining quality being discussed under various headings, and made suggestions for improving the quality of raw sugar for the refiners. Also in 1969, Saint Antoine reviewed the measures adopted in Mauritius to improve raw sugar filterability. These included the enzymatic removal of starch, liming at boiling point, more thorough phosphate precipitation, straining clarified juice, modifications to the boiling scheme to reduce the amount of “B” massecuite and screening of sugar after centrifuging. In 1970, investigations carried out in Hawaii showed that sugar produced from burned cane was superior in colour, filterability, small crystal content and polarisation. The conclusion was that the trash and soil in unburned cane adversely affected sugar quality. Tilbury reported further work on the problem of sour cane and concluded that chopped cane should be processed within 15-18 hours of harvesting. A rapid simple haze analysis method was developed to detect deteriorated cane and it was demonstrated that the addition of 3 units of dextranase to 100 ml of mixed juice removed 68.5% of the dextran in 10 minutes at 40°C and natural juice pH. Matic in 1971 described two techniques developed to improve the quality of South African export sugar. The first was to decrease the starch content of the

sugar by treatment with about 5 p.p.m. of a bacterial amylase, the second was to reduce excessive colour formation on storage by storing only very high pol sugar. During loading for export the sugar was coated with high test molasses to give the specified purity. Douwes-Dekker also in 1971 listed the seven characteristics which he considered should be used to judge the quality of the raw sugar intended for refining. These were polarisation, keeping quality on storage, free-flowing properties, affinability, ash content, colour and filterability. In the same year, James and Cameron studied the effect of dextran content on raw sugar filterability using data from the South Johnstone factory in Queensland and concluded that dextran content was a useful indicator of raw sugar filterability. Tilbury discussed the causes, effects and possible methods of control of dextran formation during sugar manufacture in Jamaica. The development of Leuconostoc mesenteroides in stored cane was accompanied by increases in the dextran content and viscosity of raw juice; a compound which was thought to differ from true dextran was frequently detected in fresh cane by the haze dilution method. Treatment of mixed juice with dextranase significantly reduced the dextran content. Farber and Carpenter reported on work concerning the identification of cane pigments that persist into refined sugar. In 1972, Alexander published details of the steps taken to improve and standardise the quality of raw sugar exported from South Africa. Murray discussed the influence of starch and insoluble suspended matter on the filtering quality of raw sugar. He concluded that suspended solids did affect filterability but were not normally present in sufficient quantity for the effect to be important. However, the presence of 150 p.p.m. of starch had a significant effect on filtration and 500 p.p.m. had a very pronounced effect. The distribution of amylase and amylopectin within the carbonate crystal matrix may influence the physical properties of the filter cake.

B. The International Commission for Uniform Methods of Sugar Analysis (ICUMSA)

The refining quality of raw cane sugar has been a subject for discussion by ICUMSA since its eighth meeting in 1932, though at that time it was simply proposed and accepted that no report be made. It must be understood that ICUMSA is primarily concerned with the standardisation of tests for the evaluation of the refining qualities of raw cane sugar; important early papers were presented to the Ninth Session in 1936 by Harman and Honig. Harman suggested tests to evaluate raw sugar; Honig arrived at similar conclusions, additionally specified a desirable crystal size (0.8-1.2 mm), and stated that a good raw cane sugar should not contain more than 0.10% of ash after losing 15% by affination. This ash should contain not more than 15% SiO₂, 2% Fe₂O₃, and 10% CaO. The Commission did not hold its tenth session till 1949, at which the referee, H. I. Knowles, summarised the refining qualities of raw cane sugar of interest to the refiner. These were:

RAW CANE SUGAR QUALITY

(1) Grist or crystal size
(2) Crystal purity
(3) Water-insoluble matter
(4) Filterability
(5) Colour
(6) Soluble ash
(7) Objectionable products of sugar deterioration
(8) Physical condition, such as hard caked sugar
(9) Amount of scale-forming constituents
(10) Substances that escape clarification and cause haze in char filtered liquors.

Tests were proposed for evaluating the first six of these, but the report concluded that there was no satisfactory evidence that any of the methods proposed would give reliable and reproducible results when applied by different individuals to a variety of raw sugars. The Eleventh Session in 1954 went little further. It was recommended that work be done on the Elliott test for filterability and that the determination of insoluble matter be further investigated especially by the centrifuging test outlined by Douwes-Dekker. Methods were also proposed for the determination of the grist of affined raw sugars. In 1958 (Twelfth Session), the referee, C. W. Davis, commented on some associate referees' views that as filterability tests on raw sugars have little value for predicting the factory filter station performance, work should therefore be carried out to supplement knowledge of the filtration-impeding constituents of raw sugar. This would be done by developing or improving analytical methods for identifying and measuring such components and by studying their behaviour in factories. At the same meeting, two new laboratory filtration systems were described; the first was developed at the Plaistow Wharf refinery of Tate and Lyle and the second originated in the laboratory of the Colonial Sugar Refining Company, following work by Nicholson and Horsley. At the Thirteenth Session, the referee expressed the hope that more work would be done towards demonstrating the relevance of laboratory tests to factory performance. This viewpoint was very evident in the recommendations proposed, of which 1-4 were adopted:

(1) That the value of filterability tests in predicting refinery performance be demonstrated over the range of conditions occurring in practice.
(2) That the gristing procedures in use be similarly examined.
(3) That additional tests for filterability and grist be sought and tested.
(4) That attempts be made to name the specific impurities in raw sugar that have marked effects on the clarification processes.
(5) That workers in this field be encouraged to publish their results in the technical press.

At the Fourteenth Session in 1966, the emphasis was again put by the referee on the need to produce laboratory tests for sugars that quantitatively

predict refinery performance, though his report\textsuperscript{5} consisted largely of a summary of work being carried out by other interested bodies. In this connection, the ideas of the International Society of Sugar Cane Technologists discussed at their Congress in 1965 are of interest. At this Congress, raw sugar quality was chosen as the subject on which the manufacturing section should focus attention in seeking to make research advances. Several aspects of raw sugar quality were listed:

1. Total impurities
2. Grist
3. Inclusions
4. Colour
5. Multivalent anions
6. Ash (particularly heavy metals)
7. Substances of high molecular weight
8. Fine suspended matter
9. Micro-organisms
10. Filterability
11. Caking and compaction.

Particular attention was drawn to crystal quality—shape, size and inclusions.

The conclusion of the referee was that work so far published or made available to ICUMSA did not establish definitive relationships between refinery performances and tests made on either raw or affined cane sugar; the recommendations finally adopted were similar to those of the previous session, except that it was suggested that attempts be made to select more specific tests for comparing with refinery behaviour, as for example:

- specific sizes of insoluble particles,
- specific classes of colour bodies,
- specific classes of ash,
- specific classes of alcohol insolubles.

In his report to the Fifteenth Session in London in 1970, the referee\textsuperscript{2} expressed gratification that this approach appeared to be yielding increasing success. The functions of ICUMSA were said to be twofold:

1. To agree on what tests were relevant
2. To specify the manner in which the tests were to be conducted in order to be recognised as official ICUMSA methods.

Methods were described for carrying out various test procedures, and the recommendations finally adopted were largely concerned with suggestions for the study of methods for grist, starch and filterability. However, a significant last recommendation was that a copy of the referee’s report, together with the discussion and final recommendations, should be sent to the secretary of the International Society of Sugar Cane Technologists. At the most recent meeting

(Sixteenth Session, Ankara 1974), the referee's report emphasised the tendency towards more specific and less empirical methods of analysis, whereby an analysis for some constituent would ultimately be based on a chemical entity rather than a general property of a sugar solution containing that constituent. Work was reported on the separation of the colorants in raw sugar, and it was recommended that future research be directed initially to identifying only those colorants which pass through to the refined sugar, which are presumably the most troublesome to the refiner. Concerning starch, it was suggested that it might be desirable to try to obtain a knowledge of the effects of individual starch components on refinery processes, as it appeared that amylose significantly depressed the filtration rate of carbonated liquors, whereas amylopectin had a negligible effect. The reason for this was that amylopectin was held within the carbonate crystal matrix whilst amylose formed a layer round the crystal. Little work was reported on the problem of dextran in a refinery, though a comprehensive review, "Polysaccharides in Sugar Cane and its Products", had been written by Imrie and Tilbury. In this, the authors concluded that soluble polysaccharides could cause poor clarification and filtration, a reduction in the rate of crystallisation of sucrose, elongation of the c-axis of the crystal, with consequent inefficient separation and purging in the centrifugals and general reduction in the economic efficiency of the mill and subsequent refining.

Regarding affination and gristing of raw sugars, several modifications to the procedure of the Hawaiian Sugar Planters Association were suggested and mention was made of a sizing method in which each crystal is individually weighed on a sensitive electrobalance. However, it was thought that, though the method would provide more meaningful information on crystal size and distribution than conventional gristing, the instrument was basically a research tool which would be unsuitable for routine monitoring of raw sugar quality. Amongst other final recommendations, it was suggested that work should continue on the identification of sugar colorants and an effort be made to establish the relative importance of amylose and amylopectin in the filtration of carbonation mud.

C. Cane Sugar Refining Research Project

This body, centred in New Orleans at the Southern Marketing and Nutrition Research Division of the United States Department of Agriculture, currently under the direction of Dr. F. G. Carpenter, is concerned with research into the problems which beset sugar refiners. Technical Sessions are held biennially when members meet to discuss these problems; not all papers are presented by members of the project team but they are all concerned with refining rather than with mill problems. At the 1966 Session, papers were presented on the significance of polysaccharide-producing bacteria in sugar refining and on laboratory filtration tests designed to predict refinery performance. In the latter, a filterability test based on the so-called plugging value was
described by Faviell and Blankenbach. This is based on the assumption that the total volume or weight of a solution which would pass through a standard filter before total plugging occurred would provide a simple means of characterising raw sugar. However, though comparison between laboratory tests and plant performance showed some correlation, it was too early to draw any conclusions regarding a definite relationship. South African workers presented a paper at the same meeting, comparing laboratory filter tests on affined raws with the filterability of factory carbonated liquors. They concluded that the most effective way of predicting the performance of a sugar in a carbonatation factory is to measure the filtration rate of carbonated liquor derived from that sugar, and that laboratory tests should be devised which duplicate as nearly as possible the conditions in any particular refinery.

At the 1968 Technical Session, three papers were given in a symposium on raw sugar quality standards. The first, by W. R. Tuson of the Colonial Sugar Company, discussed the views held in a phosphatation refinery where the question of good or bad filterability does not arise. Important points were the amount of insolubles present and the amount of scale-forming sulphate in the sugar. So far as white pans were concerned, a limit of 400 p.p.m. in the washed sugar was suggested. However, it was thought that the most important characteristic of raw sugar was good affinability. Harrison, discussing raw sugar produced in Louisiana for the Supreme Sugar Refinery, said that, following the deterioration of sugar quality from the mills within the state, it was agreed in 1966 that sugar would be purchased under specified penalties for invert, temperature and grain size. Ash was not included because it was not considered a problem in Louisiana sugar. Petri, of Godchaux-Henderson, discussed the tests applied at Reserve refinery to the raw sugar input. In addition to the normal test procedures, these included determinations of pH, crystal size, starch, sediment, filterability and ash on the affined sugar. The “expected” molasses analysis was also determined using the Tate and Lyle 1962 formula. From this the Ideal Conversion could be worked out; that is, the amount of raw sugar that would be required to make 100 pounds of refined sugar if the pol of the raw sugar was 96° and if the raw sugar was ideally refined with no resulting undetermined sucrose loss.

P. F. Meads discussed work carried out in Hawaii and in the Crockett laboratory and C. W. Davis referred to the value to both sides of the industry of group meetings to discuss mutual problems of this nature. It was necessary to make sure that lack of financial backing is not a deterrent to further research on such important matters as the inclusion of impurities in sugar crystals.

At the meeting held in 1970, no further work on quality standards was reported; the most important papers from the refineries’ point of view were those concerned with the identification of sugar colorants. At the 1972 Session, important work on the removal of trace elements in the refining processes and the amounts found residually in white sugars was presented by Clarke and Pommez. Though limiting standards for the presence of various trace elements in white sugar have been in use for many years, their presence in
raw sugar and the effectiveness of their removal by the refining processes had hitherto received little attention. The importance of the level of these trace elements in raw sugar will be discussed in a later section.

D. Sugar Industry Technicians

An important paper by Beal on methods of raw sugar analysis for evaluation of refining quality was presented at the 1963 meeting of this body. This concerned Hawaiian raw sugar destined for the C. & H. Refinery in California. Tests which had been relied on were primarily crystal colour and filterability by the Elliott test, supplemented, when further information was required, by wet screening for grain size and decolorisation tests for ease of decolorisation. It was reported that sugars with light crystal colours and high filterabilities were easy to refine and the converse was also the case. The paper described the new affination procedure which had been devised and the design of a new type of filtration test which proved more convenient to operate than the Elliott method. Colour readings were made in a Beckman Sugar Colorimeter at 420 nm in a 1 cm cell. At the same meeting, a paper by Honig and Chen described measures being taken in Peru to standardise Peruvian raws for export to the American market. It was reported that the Peruvian sugar industry was preparing a manual for the analytical methods to be followed by the evaluation of raw sugars and specifically of washed raw sugars, and desirable standards for accuracy and reproducibility of reported data were quoted. This second paper was followed up at the 1964 Session by the same authors, when they reported the result of work carried out on samples of Peruvian sugars from the 1963 crop. Factors affecting affinability and the differences between the removal of various impurities in affination were discussed. The importance of the ash level as an evaluating factor received attention and it was concluded that the composition of ash is more important than its quantity. At the same meeting, a symposium discussed raw sugar characteristics that influence refining. Main factors considered were grain size, colour, filterability, ash content, starch, alcohol insolubles, floc-forming constituents and water. The raw sugar quality standards introduced by the (then) American Sugar Company were discussed by Culp and Hageney at the 1967 Session. The established quoted ranges were:

**Whole raw sugar**
- Moisture: Factor of safety Range 0–0.28
- Sulphated ash: Fraction of non-sucrose solids 0.16–0.30

**Affined raw sugar**
- Grain size: % through 28 Tyler mesh 20–55
- Colour: ICUMSA 1958 Method 2 160–210
- Filterability: ml/10 min 50–125

*Sugar Technol. Rev., 3 (1975) 69–126*
The development of these tests and of analytical methods to measure them were described and, after an examination of many individual raw sugars, it was concluded that most raw sugars fall within the acceptable quality range.

At the 1967 Session, there was a further symposium on raw sugar standards, the points discussed being affination, moisture, ash, grain size, colour and filterability. Discussion also concerned the best way of sampling the raw sugar required for carrying out the various tests. The significance of the ash level and its effect upon various refinery operations received considerable attention, otherwise the symposium was largely concerned with the tightening up of analytical procedures. Following this, in 1969, Binkley reported the results of work carried out in the New York Sugar Trade Laboratory on contract raw sugars.

E. International Society of Sugar Cane Technologists

This body is closely concerned with every aspect of the growth of sugar cane and its utilisation as far as the production of raw sugar. Its three-yearly sessions are very widely attended—more so in numbers than any other body connected with sugar—and among the many papers on agriculture, plant breeding, entomology etc. there are normally some dealing with the general properties of the end product, rather than with the methods of producing it.

Nicholson, at the 1959 Session, discussed results obtained over several years on the presence and effects of starch in Australian raws and concluded that, though starch has a deleterious effect on filtration, it is not a major one. Only the retrograded starch fraction has any appreciable effect, that is, that portion in the form of microscopic gels. Care must, therefore, be taken in assessing the results obtained by estimating starch colorimetrically, since it is chiefly the harmless soluble fraction which is being measured.

At the same meeting there was some discussion on the observation that sugars boiled from cane juice in a laboratory pan contained less starch than sugars boiled from the same juice in the factory. No positive reason was given for this, but it serves as an illustration of the care which must be taken in trying to relate laboratory results to those from the factory or refinery. At the 1962 Session, Antoine et al. of the Mauritius Sugar Industry Research Institute presented a paper which demonstrated most strongly the serious effects which can arise from osmophilic yeast contamination of raw sugars. The work was largely concerned with an investigation into the presence of these yeasts in Mauritian raws, which had been sparked off by the Mauritius Sugar Syndicate after its having been informed by Tate and Lyle in 1960 that, on emptying a large silo in which 4300 tons of Mauritian raws had been stored for several months, it was found that the sugar had deteriorated badly. The polarisation of the sugar had dropped by 2.7° during storage. Bacterial examination revealed the presence of two distinct types of yeast. One was of the normal osmophilic type commonly encountered; the other, which was present in considerable numbers, was allied to brewing yeast, contained invertase, was highly ferme-
tative and had not been seen in raw sugars before. The results of the investigation demonstrated that osmophilic yeasts were present in sugar from all the Mauritian factories, but probably only a few of the types found could prove potentially damaging. The yeasts are capable of growing with extreme rapidity under favourable conditions, causing rapid decomposition of raw sugar. Yeasts present in sugar products in the early stages of manufacture are destroyed in the normal boiling processes, and their presence in the raw sugar was (in the case of Mauritius) reported to be due to post-contamination of the massecuite in the crystalliser. To improve the keeping quality of the sugar, steps should be taken at the factory to prevent or reduce considerably the risk of this contamination.

Douwes-Dekker presented a paper on raw sugar filterability and described attempts being made in South Africa to reduce the starch content of raw sugar. Three methods were being tried:

1. Reducing the starch content of the cane.
2. Removing the starch from mixed juice using the method of Nicholson and Horsley. This is based on the fact that, if mixed juice is kept at the optimum pH and temperature for the right period of time, the starch-destroying enzymes naturally present will remove a large proportion of the starch.
3. The use of a centrifugal separator.

In 1965, Moritsugu and Payne of the Hawaiian Sugar Planters' Association Experimental Station presented a paper which studied the effects of circulation rate, ash-concentration, degree of supersaturation, temperature level and crystal size on crystal quality, and concluded that high circulation rates were beneficial, but a faster rate of crystallisation, achieved by increasing the sucrose concentration, gave poorer quality crystals. High temperatures produced crystals of poorer colour and higher ash, and the level of included ash increased with the size of the crystal. Recrystallisation did not greatly improve pol, filterability or crystal colour.

Tu reported work carried out on the insoluble matter in sugar crystals which has a major effect on filterability. The particles have an average dimension of 0.9 μm with a significant proportion in the range 0.3–0.4 μm. They contain lipids and principally low molecular weight substances along with higher molecular weight nitrogen- and methoxyl-containing compounds.

At the same meeting, there were quoted the standards then in effect which governed the sugar being supplied from Hawaii to the Crockett refinery. These were:

- Pol: 97.5–98.8%
- Grist: grain passing through a Tyler 28 sieve not to exceed 34%
- Filterability: 220 under standard Hawaiian test
- Colour: 6 units (each unit is ten times the attenuation index at 420 nm)
- Ash: 0.75% on unwashed sugar.

In 1968, at a symposium on sugar quality, Meads presented an important
paper on the work started in Hawaii in 1956 to minimise refining costs at Crockett. The programme then initiated included a premium system to provide increased payment for higher quality raws. To demonstrate the effect that certain factors in raw sugar can have on its refining characteristics, two raw sugars were carefully compared. Their properties were:

<table>
<thead>
<tr>
<th></th>
<th>Colour</th>
<th>*St</th>
<th>Elliott Crystal filterability</th>
<th>Thro' 30 screen</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sugar A</td>
<td>97.9</td>
<td>35.1</td>
<td>3.8</td>
<td>86</td>
</tr>
<tr>
<td>Sugar B</td>
<td>97.9</td>
<td>34.9</td>
<td>5.5</td>
<td>66</td>
</tr>
</tbody>
</table>

The effects of Sugar B as compared with Sugar A were:

- Melting rate: down 28%
- Filter aid used: up 72%
- Char burned: up 73%
- Colour of white sugar: up 62%

The work had shown that, apart from the damaging effect of fine suspended matter in filtration, there was good correlation between starch content and filterability, and that the colour which was most readily removed in the refining process was sensitive to pH and low in molecular weight.

Methods of measuring the important characteristics of raw sugar had been reviewed. Colour, filterability and particle size measurements were made on an affined sample; it was concluded that the methods currently in use for colour, filterability and grain size were unsatisfactory; those for ash and pol were acceptable. No definite indication was found as to the refinery product on which filterability should be determined, though that on washed raw sugar seemed to have broader application than those on carbonatated or phosphate-treated washed sugar liquor.

To produce a schedule of premiums it would be necessary to estimate the raw sugar quality that would provide the required output of refined sugar at the desired quality level in the existing facilities at normal refining costs. Normal costs would exclude extra washing and other costs at the affination station resulting from fine grained sugar, costs of extra filter aid, supplementary decolorisation and cost of reprocessing any remelt sugar.

Crockett established base qualities for each of five factors: pol, ash, colour, filterability and grist (the first two were later dropped from the premium schedule as they became always acceptable). Refining experience for the previous year was then reviewed and costs related to actual quality of raw processed, together with any extra costs. The total costs furnished the basis for the schedule of premium allowances.

Also at the 1968 Session, a paper was presented by Yamane et al. on the effects of impurities included in affined sugar. They identified three groups of melanoidin colorants varying in molecular size, those of largest molecular size being most easily removed. They also postulated that high colour in affined

sugar meant high colour in the decolorised liquor, and that insoluble impurities have the highest filtration-retarding effect on melter liquor, while with carbonatation liquor slurries, difficulties are due to starch and the fact that filtration-retarding impurities are occluded and, therefore, cannot be removed by washing in the affination process.

At the 1971 Session, Matic discussed the colorimetric determination of starch in raw sugar. Tilbury presented a paper on the formation of dextran in Jamaica raws and Farber and Carpenter discussed the identification of cane pigments that persist into refined sugar. Cane pigments are one of three classes of cane sugar colorants, the others being caramels and melanoidins. Some of these pigments are not removed in the refining processes and appear in the refined sugar. Using high voltage paper electrophoresis and chromatographic methods, some of these pigments have been identified. They are chlorogenic acid, caffeic acid, p-hydroxycinnamic acid, 4-hydroxy-3-methoxycinnamic acid, 4-hydroxy-3,5-dimethoxycinnamic acid, kaempferol and umbelliferone.

III. THE PRESENT SITUATION

It is very evident from the lengthy preceding historical survey that an enormous amount of work has been carried out over many years in an effort to identify the factors affecting the refinability of a raw sugar and to try to eliminate those which have an adverse effect. Certainly, some advance has been made but whether its value is commensurate with the amount of time and labour expended seems unlikely. There are various reasons for this, but probably the most important is a consequence of the diversity of sugar refining processes in use, so that a raw sugar producer would probably have to tailor his product to the requirement of almost every individual refiner. There are, of course, some general rules which apply, e.g. a raw sugar must affine well and it must be possible to filter readily a solution of the affined sugar, whether carbonatated or otherwise treated.

All sugar refiners use an initial affination process (though even in this there is no complete agreement as to the best way of carrying it out), but after that the variations start. For defecation there are two principal systems: carbonatation where good filterability is of prime importance and phosphatation where it is much less. It must be said, however, that several leading refineries do not use a defecation process at all, the addition of filter aid and filtration being considered sufficient, though to enable them to do this, they must have a guaranteed supply of high test raws. The increasing importance and popularity of the Talofloc process must also be taken into account in this context.

Decolorisation can be carried out by the classical method using bone charcoal, granular carbon of high decolorising activity, powdered carbon on a throw-away basis, or ion-exchange resins. All systems have their strengths and weaknesses; for example, bone char needs a large installation, but in addition to removing colour, it removes some ash from sugar liquors; this can be an
important consideration. Granular carbon, which because of its high decolorising power needs a much smaller plant, does not remove ash.

The practices of white sugar boiling are fairly standard throughout the sugar world; what is not is the means of disposal of the last white jet which contains the concentrated impurities originally brought into the white sugar boiling station. This may be used for the production of soft brown sugars or of saleable syrups for domestic consumption. For refineries which do not produce either, it is sent back to the recovery (remelt) station.

The disposal of the affination syrup produced in the first stage of the refining process can be either to recover the sugar, leaving as a final product well-exhausted molasses, or (if the quantity is small enough) to use a carbonation and filtration process for the production of soft brown sugars or table syrups.

It will be clear from the foregoing that any refinery should ideally be able to standardise on one type of raw sugar, and in certain instances this is possible, the best known case being that of the California and Hawaiian Company. However, the less fortunate producer with differing markets could find himself in the position of having to supply a different sugar for each one. Even within the same market, seasonal variations can occur; for example, at the Liverpool refinery of Tate and Lyle there is normally a much larger demand for soft brown sugars during the winter months than during the summer months. This means that the input of impurities into the refinery can safely be higher in the winter than in summer. That (as mentioned earlier) the exact opposite used to occur until fairly recently was unfortunate but true.

The processes of sugar refining will be described in more detail later, but it is now possible, following the review of published literature, to list those factors which can have an influence on the sugar refining process and even possibly to add one or two which have not yet received much attention. The list has grown considerably over the years and is likely to keep on growing as the white sugar users become increasingly critical in their specifications, so that the refinery will need to become increasingly critical of the producer, who will need to accept the same constraints as the refiner.

A difficulty is that a refinery using a normal control laboratory may not be able to carry out all the tests on a raw sugar completely to assess its refinability; also, the refiner may need to take the raw sugar into the refinery as soon as it is delivered. In this case, the sugar might be melted before any tests could be made; retrospective tests would no doubt help to decide whether the sugar was within specification or not, but would be too late to give the refiner any indication of what particular problems might arise with that particular melt.

The following is a list of the factors which might be considered to play a part in assessing the refinability of a raw sugar:

1. Polarisation
2. Moisture content
3. Safety factor (or dilution indicator)
4. Total ash

(5) Sulphate content
(6) Polyvalent anions
(7) Colour—particularly colour which persists into refined sugar
(8) Affinability
(9) Filterability
(10) Starch content
(11) Polysaccharide content (other than starch)
(12) Insoluble foreign matter
(13) Bacterial count
(14) Trace elements
(15) Insecticides and herbicides
(16) Silica
(17) Floc-forming constituents.

This is a formidable list, and it will be necessary to discuss the sugar refining processes in more detail to determine how each individual factor can play its part in affecting some portion of the process.

IV. THE SUGAR REFINING PROCESS

Sugar refining as normally practised can conveniently be broken down into several unit operations. These are:

A. Delivery and storage
B. Affination
C. Defecation
D. Filtration
E. Decolorisation
F. Crystallisation
G. Recovery
H. By-products

It will be necessary to consider these separately and show how the various factors listed previously affect each of them.

A. Delivery and storage

Raw sugar is now normally delivered to the refiner in bulk; as a consequence, one of the most troublesome of former nuisances has disappeared: that is, the arrival of sugar in bags at the refinery in the form of solid blocks caused by compression and drying of the sugar during transit. This was probably a result of the sugar being packed too hot. The arrival of sugar in this form presented a severe challenge at the refinery; not only was there a great problem in getting it out of the bag, but the blocks had to be broken up manually before it was possible to do anything with them. Sugar in bulk has been of great benefit to the refiner in a variety of ways, but there are problems concerned with the

storage of large quantities of sugar in this form. They are mainly those concerned with microbiological deterioration and colour formation.

Destruction of sugar by osmophilic yeasts can take place in the syrup film surrounding the crystal providing conditions are right, that is, if the syrup film is sufficiently dilute. To decide whether the sugar is safe to store, a so-called safety factor (S.F.) has been devised (originally by the Colonial Sugar Refining Co.) which relates the water content of the sugar to the impurities present and is expressed by the formula:

\[ \text{S.F.} = \frac{W}{100 - S} \]

where \( W \) is the water content of the sugar and \( S \) is the polarisation. The safe limit for this was originally fixed at a figure of 0.333 or less, but this might be considered too high by some and an upper limit of 0.25 has been widely expressed as being more satisfactory, the Amstar Corporation, for example, fixing a limit of 0.28. However, the figure decided upon by any individual refiner will depend to some extent upon the conditions, climatic or otherwise, under which he intends to store the sugar. If the figure is above the upper limit then the sugar cannot be stored without danger of quite substantial deterioration and sugar loss; thus any lot of sugar arriving at a refinery should be tested immediately before its destination is decided: if the safety factor is too high then the sugar should be melted immediately. However, this is the ideal policy which in practice it is not always possible to carry out, and the refiner is largely dependent upon the producer, who probably has a direct interest himself, as large quantities of sugar might have to be stored at a sugar terminal.

A modification of the safety factor has now come into use. This is called the Dilution Indicator (D.I.) and is expressed by the formula:

\[ \text{D.I.} = \frac{100 \times \text{Moisture}}{\text{Non-pol solids}} \]

A safety factor of 0.25 corresponds to a dilution indicator of 33 and a safety factor of 0.333 corresponds to a dilution indicator of 50. Thus 50 might be considered the safe upper limit.

A further method of assessing the probable keeping quality of a raw sugar has been advocated by Powers. This is a measurement of the Equilibrium Relative Humidity (E.R.H.), which is defined as the relative humidity existing when equilibrium is reached between the syrup film on the crystal and the surrounding atmosphere. Methods of measuring the E.R.H. are suggested and a relatively simple spot check devised by Davis is described. Further work on E.R.H. has since been reported, but there appears to have been no general acceptance of the method and interest recently has waned.

The second factor concerned directly with the safe storage of raw sugar in bulk is that of temperature of storage. Though (as far as can be ascertained) spontaneous overheating in raw sugars to the point of combustion is unknown, temperature increases in stored raw sugar have been noted. This leads to
excessive colour formation and increased sugar destruction owing to an increase in osmophilic yeast activity. A maximum safe storage temperature of 38°C (100°F) has been suggested by Clarne; elsewhere a figure as low as 25 ± 3°C (77 ± 5°F) has been recommended.

The third problem involved with the safe storage of raw sugar in bulk is that of determining what is a safe upper limit for the osmophilic yeast count. What this is will, of course, depend upon the safety factor level. However, it has been reported that raw cane sugar has been safely stored for periods up to 20 months and at depths of 70–80 feet under the following maximum conditions:

Safety factor: 0.25  
Osmophilic yeast count: 10,000  
Temperature: 25°C (77°F)

B. Affination

The affination process is designed to remove as completely as possible the impurities in the syrup film surrounding the sugar crystal in raw sugar which contains the bulk of the impurities. Of course, included impurities and colour are inaccessible to affination, so the effectiveness of this unit process is determined by the proportion of included and occluded impurities. With good affination it is likely that the impurity level in the affined sugar will be about 15% of that in the whole sugar. The process normally consists of mingling the raw sugar with affination syrup (already produced in the process) to a so-called magma. Usually the magma will contain about 10% of water and will be warmed, partly to reduce the viscosity of the syrup film, so making it more readily removable, and partly to enable the magma itself to flow more readily to the centrifugal machines. However, opinions differ as to the correct temperature and how this temperature should be achieved: whether by heating the magma itself as with a Stevens coil, or by heating the returned affination syrup prior to mingling. However, the magma temperature is usually in the region of 40–50°C (104–122°F) and the time allowed for completion of the mingling process is about 30 minutes.

The magma is then fed to centrifugal machines where it is spun to remove the mingling syrup containing the raw sugar impurities, the process being completed by following up the initial spinning with a water wash. Part of the syrup spun off is returned for mingling and this should ideally be that portion produced by the application of the wash water, though separation of the “green” syrup from the wash is not always practicable.

It will be clear from the foregoing that affination is the unit process which removes by far the greatest proportion of impurities in the original raw sugar—about 85%—and anything that reduces its efficiency can lead to subsequent difficulties. It is uneconomic to try to remove at other stages of the process impurities which should have been removed in affination. To refine

sugar successfully one must start correctly; a maxim which has been preached consistently by one of the authors during his years in a sugar refinery, he hopes with some success.

There are three factors which can adversely affect the efficiency and effectiveness of the affination process. These are:

1. The level of impurities in the whole raw sugar
2. The quality of the grain
3. The presence of finely divided insoluble impurities—fibres, etc.

If a minimum purity is part of a contractual agreement then the first should be taken care of; there will perhaps be a need to use more wash water on the centrifugal machines for sugars at the minimum end of the purity scale than for those of higher purity.

The most important factor in obtaining good affination is the quality of the raw sugar grain. If there are many fine crystals present, these can “blind” the sugar wall in the centrifugals by blocking of the channels between the crystals. This results in inefficient purging and washing, with the probable use of more wash water and a significant decrease in the quality of the affined sugar. In extreme cases where the quantity of fines is very high, it is possible for purging and washing to stop completely; any wash water applied might stay on the surface of the sugar wall in the centrifugal machines to run off when the machine comes to rest. Fortunately, this extreme condition is in our experience extremely rare, but there is a very large number of situations possible between extremely bad and extremely good affination. Even mixing sugars of two different origins or lots can have a damaging effect even though individually each may affine quite well.

The importance to a refiner of receiving raw sugar with minimum spread of crystal size, which itself should not be too small, will then be obvious; grist analysis of the affined raw sugar might be part of a contractual agreement. As small grain produced in the raw sugar boiling operation cannot be removed by screening as it can in the case of refined sugar, good control of the pan boiling is essential. It is also important not to damage the crystal after it has been produced; the broken crystals typical of the earlier continuous centrifugal machines could be a highly potent cause of bad affination. This has been recognised by the development of the wide casing machine.

Finely divided insoluble impurities present in the raw sugar can act in the same way as fine grain, i.e. causing poor affination by blinding of the sugar wall. They can also have a subsidiary effect by blocking syrup gutters and pipes. This might cause an overflow of low quality affination syrup into the affined sugar.

C. Defecation

The defecation process as normally practised is intended to remove colloidal impurities which largely contribute to the colour of the affined sugar, together with non-coloured colloids such as starch and gums. The two standard
methods are carbonatation and phosphatation, though some refineries use only lime and an added filter aid or in extreme cases only filter aid followed by filtration. These latter practices can only be acceptable when the affined raw sugar is sufficiently good in colour, or there is a very large decolorisation installation relative to throughput.

The carbonatation process might be expected to remove about 50% of the residual colour. The process depends on the addition of lime to a solution of the washed raw sugar and the precipitation of this as calcium carbonate by gassing with carbon dioxide normally derived from washed boiler flue gas. The precipitation carries with it impurities which are removed by filtration, the calcium carbonate playing a second role as a filter aid.

In the phosphatation process, lime and phosphoric acid are added to the sugar solution. This forms a precipitate of calcium phosphate that performs the same function of trapping impurities as does the precipitate formed in the carbonatation process. In phosphatation, as normally practised, this precipitate is not removed by filtration. The Williamson system uses a clarifier and air flotation; the scum remains on the surface of the sugar solution and is continuously removed mechanically. Colour removal during the phosphatation process may be 20–40%.

Coming increasingly into prominence is a new method of defecation, the so-called Talofloc process developed at the research laboratories of Tate & Lyle Limited. This depends upon the use of a cationic surfactant to precipitate colouring matter and other high molecular weight anionic impurities from solutions of affined sugar. The colour is precipitated in the form of very small particles which can be removed by filtration but only at rates which would be unacceptably slow in a refinery. The process is, therefore, of most benefit where a filtration stage is not required (excepting perhaps a polish filtration); that is, in the standard phosphatation flotation process. In this it has been found that the addition of a suitable flotation aid brings about the rapid and complete separation of flocculated impurity. The flotation aid is an anionic polymer developed specifically for use in the Talofloc process. The colour removed by the process depends on the amount of Talofloc added, but at the recommended rates of usage is between 50 and 75%.

It will be clear that the defecation processes themselves will not be affected by the quality of the solution of affined raw sugar being treated; what will be affected is the effectiveness of the process in removing residual undesirable impurities. In carbonatation, for example, only a small portion of the lime added takes part in the carbonatation process as such; the remainder provides the calcium carbonate filter aid. It follows that the use of more lime would have no benefit in removing more colloidal impurities and colour (though it might help filtration). In order to ensure, therefore, that the minimum amount of impurities goes through to the decolorisation process, it is necessary to ensure that the minimum goes to the defecation process; such arguments apply to the phosphatation and Talofloc processes also, though in the case of Talofloc it is technically feasible to improve the effluent colour by the addition of surfactant in quantities greater than are economically attractive.

D. Filtration

The precipitates formed in the defecation process can be removed in two ways: either by filtration, as in the simple defecation processes and carbonation and some phosphatation processes, or by such means as flotation and mechanical removal as in phosphatation flotation processes and the Talofloc process. In the latter, all that may be necessary is a polish filtration of the effluent.

However, it is clear that filtration is one of the major unit processes used in sugar refining, and it is likely that more thought and time has been expended on trying to improve the filtering qualities of raw cane sugar than on anything else. A great deal of effort has also been expended on attempts to improve the filter itself, and these come now in great variety of styles, shapes and sizes. However, though some may have certain advantages over others, it remains true that what decides how good or bad filtration will be is the characteristics of the material being fed to the filter. These are decided by:

1. The nature and quantity of the filtration-impeding impurities present.
2. The efficiency with which the preceding defecation process has been carried out.

The nature of the filtration-impeding impurities and proposed methods for their reduction in raw cane sugar have received considerable attention for many years and many efforts have been made, particularly for carbonation processes, to relate laboratory carbonation and filtration tests to results that one might expect to get on a plant scale. However, like many other things straightforward in theory, this is not so easy in practice, as the carefully controlled conditions of the laboratory are not so easily reproducible on the plant. Some success in doing this has, nevertheless, been reported, but most laboratory work has been confined to testing solutions of affined raw sugar using various testing techniques and endeavouring to relate this to the expected refinery results. As a perhaps irrelevant sideline on this, it has long been noted by one of us that we might expect trouble in filtration with raw sugars that filtered slowly during polarisation filtration. Perhaps our test procedures are becoming too complicated?

The main filtration-impeding materials present in whole raw sugar and to a lesser extent in affined raw sugar are polysaccharides such as starch and dextran. Concerning the former, recent work has suggested that the important component of starch affecting filtration is the amylose; this forms a layer round the crystals in carbonated slurries whilst the amylpectin is held within the crystal matrix. Dextran and other soluble polysaccharides give juices of high viscosity in the mills leading to poor clarification and filtration, which in turn leads to a reduction in the economic efficiency of the refining process. However, little effort seems to have been made up till now in attempting to quantify the effect of dextran on refinery processes or at least to publish any work on this. Some effects known to the authors will be dealt with later.

Various methods have been tried to reduce the starch content of raw sugars, including centrifuging the expressed juice and vacuum clarification using the Rabe process. However, the most successful and economically attractive method seems to be the use of bacterial enzymes. Because these are invertase-free, or virtually so, the method can be used without the danger of any associated sucrose loss. Minimising the quantity of dextran in the expressed cane juice and the sugar prepared from it depends largely on the immediate use of the cut cane. In cane cut for any length of time and left lying without crushing there is a rapid increase in the dextran content; the upper limit of the safe keeping time for cut cane appears to be about 15 hours.

Filtration is also affected adversely by the presence of measurable amounts of silica. This appears to act by gradually and progressively blocking the apertures in the filter cloth. If there have been marked quantities of silica in the material being filtered, a portion of the cotton filter cloth, when acid-cleaned to remove calcium carbonate and then ignited, will display a silica lattice showing where the silica has become attached to the fibres.

Filtration can, of course, also be affected by the presence of excessive quantities of finely divided insoluble matter. These, originating from the fibrous material of the cane or from the soil, act by blocking the filtering surfaces. Furthermore, filtration can be affected by mismanagement of the defecation processes. This has nothing to do with the raw sugar being melted, but it may be of interest and useful to note here the result of work carried out by Bennett which was put into application at the Liverpool refinery, using a carbonatation process, in 1964. At that time the melting of certain cargoes of Australian and Mauritius raws had resulted in the melting rate being restricted to a figure well below that necessary. Bennett's work showed that to achieve the optimum filtration results some changes in the carbonatation procedure would be necessary. There should preferably be a period of time allowed between the addition of lime to the melter liquor and the start of gassing, though this time need only be very short. The gassing operations ought to be carried out at a very much higher temperature than had hitherto been thought necessary or desirable and, as far as possible, the complete gassing process, using the maximum amount of lime possible, was to be carried out in one tank (at this time Liverpool operated three tanks in series). These changes markedly improved filtration and have been standard practice since.

E. Decolorisation

The two major decolorising agents in use in the sugar refining industry are bone charcoal and granular carbon. Powdered carbon is also used, though on a very much smaller scale, while resins seem to be principally used for polishing charred liquor.

This section will concern itself, therefore, with bone charcoal and granular carbon. Bone charcoal, referred to, in England at least, as "char", has a long and proud history in the sugar industry. It has been the main decolorising agent.
in use for well over 100 years and, though the use of granular carbon is now increasing, char in some respects remains pre-eminent. Its great value is that, in addition to removing colour, it also removes ash and in so doing is probably the refineries' greatest safeguard against the chance leakage of toxic impurities into the refined products. Its disadvantage is that it requires a very large installation. Another current problem (which has nothing to do with its value in the sugar industry) is that the supply of bones for its production is becoming increasingly short. To achieve the same degree of decolorisation a granular carbon installation can be very much smaller, with consequent savings in space and in maintenance and running costs. However, it does not remove ash in significant quantity, and consequently some doubts have arisen as to its value as a "cordon sanitaire" in the same sense that char is. In view of the fact that the Liverpool refinery of Tate & Lyle operates with carbon only, this particular aspect has been the subject of much work within Tate & Lyle. Though much work remains to be done, the results obtained so far indicate that granular carbon does remove heavy metals from sugar liquors, certainly in the quantities that are normally likely to be met, but it is not so efficient at doing so as char. This is of some importance in view of the very lengthy runs which are normal with the use of granular carbon, particularly as it seems that whereas with char the removal of trace toxic impurities is virtually complete, in the case of granular carbon there is a gradual increase in the effluent concentration as the run proceeds. Thus it is important for the refiner to be assured that toxic elements are not present in his raw material to an extent that will present any problems. This has so far presented little difficulty, but will need increasing attention with the move away from char to granular carbon. There is, however, another and associated problem which arises out of the increasing use of pesticides and herbicides in the cane-growing areas. Little, if anything, is known about the fate of those compounds in the sugar refining processes or what part of the process may be relied upon to remove them. Some work is proceeding on this, but so far the levels of organochlorine or organophosphorus pesticide residues, even in refinery molasses, has been below the limits of detection or toxicity. It is not even known whether they are likely to appear in the end products in the form in which they were originally used. A great deal of work requires to be done, and that without undue delay in this matter. In the meantime, raw cane sugar producers should have in mind the fact that what they are using with benefit may have damaging effects elsewhere. There have already been various well publicised instances of the damage that can arise in some areas from the use of such materials.

However, char was originally introduced into the sugar refining industry as a colour remover, and that is its major function as it is also of granular carbon. For most of the time since its introduction, its users have been content to let it do its job without worrying too much about how it did it or what were the characteristics or identity of the colorants that they were removing (or trying to remove), though they did known by simple observation that some colours were easier to remove than others. However, this is now changing and much work is
being done on the colouring matter in raw sugars, and in particular efforts are being made to identify those which persist into refined products\textsuperscript{\textcopyright 1}. This is an important move forward; it is at least an advantage to identify the enemy before making any plans to deal with it. However, putting it in its simplest terms, the refiner clearly does not want to buy any more colour than he needs, which means that, quite apart from the naturally occurring pigments, the raw sugar producer must minimise colour development during production. This development can occur not only in the liquid phase but also in the final product due to the Maillard reaction. Raw sugar likely to be kept in store for any length of time at the terminal, or indeed the refinery, must be stored cool, certainly at a temperature not higher than 38°C (100°F).

Another factor influencing the decolorisation of sugar liquor, certainly by char, is the effect of what have been termed excess polyvalent anions. This has been defined as the difference between the number of all adsorbable or polyvalent anions in a sugar solution and all adsorbable or polyvalent cations. The implications of this work by Carpenter et al.\textsuperscript{\textcopyright 2} have been fully reported, and they suggest that in liquors where excess polyvalent ions are present, the degree of colour removal with char can be increased by the addition of a suitable calcium salt. These findings were substantiated by work in the Central Laboratory of Tate & Lyle refineries, where decolorisation over char was improved by the addition of calcium chloride to the sugar liquor. However, there is, of course, the disadvantage that the calcium addition increases the ash of the effluent liquor, with the result that, so far as the authors know, no practical use is being made of these findings. It will be necessary to tackle the problem in a fundamental way by preventing the presence of excess polyvalent anions, which means initially determining their source.

So far as granular carbon is concerned, the problem does not arise as excess polyvalent anions do not have any effect on its decolorising properties.

\textbf{F. Crystallisation}

The final stage in the refining is the crystallisation of sucrose from the decolorised liquor and the removal of the crystal from the mother syrup by centrifuging followed by drying. By this stage, the refiner hopes that the vast majority of the impurities have been removed, and indeed there should be fewer problems involved in the operation of a white boiling house than in any other part of the refining process. However, it must be appreciated that the process of crystallisation is in itself a highly efficient method of removing impurities, and thus the great bulk of those that enter the white house will be concentrated in the jet spun off the final boiling of white sugar. The level of impurities in this jet will normally decide its destination.

So far as the final product is concerned, by the time it has been crystallised from solution, washed and spun in a centrifugal machine, dried and packed, the refiner hopes that he has produced a product within his own rigid specifications and a foodstuff which is purer and more free from contamination than any

other. Not only that, he hopes to have done it economically. He must, however, be producing a material within his customers' specifications also, and these will include not only colour, invert, ash etc., but also bacterial counts and floe-forming characteristics, the latter being due to minute traces of floe-forming impurities originating in the raw sugar which have avoided removal during the refining process. He must also have produced a material that satisfies the various legal requirements governing food production, many of which are concerned with the level of trace elements in the product.

G. Recovery

The recovery (or remelt) process can be adversely affected by many of the factors already discussed relative to the other refining processes. What is more, as many of the original impurities in the whole raw sugar have been concentrated in the feed material to the recovery plant, the effect can be very great. The objective of the recovery operation is further to concentrate these impurities so that they can be removed together with a minimum of sucrose, leaving behind a relatively pure sugar for reprocessing. The effect of this concentration can be simply illustrated by experience at the Liverpool refinery of Tate & Lyle Limited, where part of the final molasses, after suitable processing, is utilised as part of a mix for making edible treacle. The legal limit for lead in such a case is 5 p.p.m. solids, which means that molasses used for edible purposes must not contain more than 3.66 p.p.m. as sold. To maintain this, the lead content of the raw sugar must not be more than a trace; even a figure as low as 0.5 p.p.m. will build up to an unacceptable level in the final molasses. Fortunately this does not often happen, but it can be very troublesome when it does. The highest lead content of molasses recorded at Liverpool is 26 p.p.m. Levels such as this present serious problems in the disposal of the molasses, and continuous attention must be given to the lead content of the raw sugar input in order to foresee trouble and try to avoid it, possibly by blending another raw sugar with a badly contaminated one.

The presence of excessive quantities of crystallisation-inhibiting materials, dextrins, starches, gums, can have a serious and very damaging effect on the efficiency of the recovery house operations. They lead to extended pan cycles due to slow crystallisation, poor exhaustion and the production of crystals that have poor centrifuging characteristics. Thus costs are increased by extra fuel consumption, excessive sucrose going out in molasses and the losses due to inefficient machining of low grade massecuities. In the extreme, the latter can lead to a slow-down of the entire refinery. There will inevitably be, in any case, excess reworking of low grade syrups which compounds the difficulties and leads to increased sugar destruction.

This is a formidable list and clearly of great importance. Unfortunately the complexity of the effects of these materials makes financial assessment very difficult indeed, though it is no doubt possible to put an approximate figure on it. However, it is safe to say that severe financial losses can be involved directly.

They can also result inadvertently by contamination of the recovery process by dextran-producing organisms, such as *Leuconostoc mesenteroides*. It is part of the recovery process function to operate as a sink, collecting and dealing with unwanted materials from other parts of the process, which include varying quantities of low brix sweet waters, the temperature and pH of which are usually conducive to the growth of more *Leuconostoc*, with consequent sugar loss. This problem can, of course, be minimised by not producing light sweet waters, though this is a policy of perfection and one much easier to carry out in a recently built refinery than it is in an old plant. The amount of lights for disposal is also affected, of course, by the particular refinery process being worked.

Sugar destruction can also occur by infection with other organisms, particularly yeasts brought in with the raw sugar. Some of the manifestations of yeast infection can sometimes be so severe as to be alarming. The safeguards against losses of this nature are the classic refining ones of keeping the place clean and not allowing quantities of light sweet water to lie around. Again, it is very difficult to quantify the actual amount of sugar destruction which is due to bacterial infection, but there is no doubt that even in the best regulated refineries this cause is responsible for a significant portion of the unknown sugar loss. Losses caused by infection with osmophilic yeasts should not normally be any problem, since recovery house syrups are generally at too high a solids concentration for them to multiply.

The next and most direct factor which affects the recovery plant operations is the actual load of impurities together with its associated sucrose that it has to deal with. This obviously depends upon the analysis of the raw sugars intake which, as has already been said for the less fortunate refineries, are not constant. Indeed, a recovery plant can be under so little pressure it is almost idle, or so busy that it can restrict the rate of melting, all within the space of a few weeks. In the latter case, the efficiency of the molasses exhaustion may well suffer. Those refineries which have a continuing supply of constant analysis high quality raws are in the most favourable position; indeed, it is possible for a refinery to operate without a recovery plant at all provided the affination syrup produced can all be used in making soft brown sugars or edible syrups. In a much less favourable position are those refineries which have no access to a standard raw and whose input can vary from week to week and almost from day to day. Under those circumstances, the recovery house has to be designed to cope with widely ranging conditions, which implies that for a major part of the year the plant will be underutilised. Thus the effects of difficulties in the recovery house are felt throughout the whole refinery. If it is very heavily loaded and the melting rate must not suffer, then it may be necessary to decrease the quality of raw sugar affination by reducing the wash on the affination machines (even though this offends against one of the cardinal rules of sugar refining, which is to "start right"). This leads to difficulties at later stages of the process and it may be considered better to overcome the difficulty by letting more sucrose go out as molasses. Either way heavy financial loss can be involved.

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H. By-products

All refineries face the problem of disposal of the last white jet, that is the syrup off the lowest grade of white sugar boiling which contains the accumulated impurities brought in with the raw sugar, other than those separated in affination syrup. Some refiners use this as a base material for the manufacture of so-called soft sugars or of edible syrup, making up the impurities required if necessary by using materials such as affination syrup. The making of these by-products is normally a relatively simple process; colour will normally present the most serious difficulty. These are usually highly coloured materials which paradoxically quite often have to meet relatively tight colour specifications. But as they contain a relatively high amount of impurities, then the same problems of concentration as exist in the recovery house (though to a lesser degree) will apply, i.e. traces of toxic materials coming in with the raw sugar could possibly rise to unacceptable levels. Thus constant examination and testing are required.

I. General

Scaling of heating surfaces can sometimes become a major problem with most refiners. The effects are observed in poorer heat transfer across the heating surfaces, increase in pan down time due to taking them out of duty more frequently for cleaning and, in extreme cases, contamination of sugar with scale. Thus, extra costs can be involved in loss of melt, extra steam consumption and cleaning costs including chemicals and labour. The particular type of scale present will depend largely on the use of the heating surface: for example, with the white pans at Liverpool refinery it is largely calcium sulphate, whereas in the evaporation of light low grade materials, the amount of silica present in the scale can be significant. Clearly, not only the actual ash level in the raw sugar but also the composition of the ash are of great significance to the refiner.

V. RAW SUGAR QUALITY STANDARDS

Though investigation into the factors affecting the refining qualities of raw cane sugar have been in progress for many years, it is only comparatively recently that any attempt has been made to utilise these investigations for their ultimate use; that is, to fix standards for raw sugar input into refineries with penalties for sugar falling outside these specifications.

Earlier, a lengthy list of the properties of raw sugar which might have some effect on its refining qualities was set out. It would certainly be impracticable at the present time to specify limits for all the properties mentioned, even if for no better reason than that of the problem of getting agreement between buyer and seller on what the limits should be and on methods of test. It is, therefore, a
matter of considering what factors have most importance and can also be measured accurately and with good reproducibility. When quality standards were first implemented, these were considered to be purity, keeping quality, ash, colour and filterability. This section will deal with some of the standards which have been laid down; this might mean some repetition of figures in previous sections but it is advantageous to collect all such information together.

In America the standards first laid down by the Amstar Corporation were as follows:

**Whole raw sugar**

<table>
<thead>
<tr>
<th>Factor</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture: Factor of safety</td>
<td>0–0.28</td>
</tr>
<tr>
<td>Sulphated ash: Fraction of non-sugar solids</td>
<td>0.16–0.30</td>
</tr>
</tbody>
</table>

**Aflined raw sugar**

- **Grain size**: % through 28 Tyler mesh 20–55
- **Colour**: ICUMSA 1958 Method 2 160–210
- **Filterability**: ml/10 min 50–125

The test employed to measure filtration was that of Coll et al. employing the use of Millipore filter Type RA at 22 in. of vacuum for 10 minutes and measuring the volume of the filtrate. It is necessary to apply a calibration factor to the result to account for variations in the filtering characteristics of the membranes being used.

Other American refineries would not of necessity use the same criteria, possibly because they did not even use the same process and possibly because they did not agree with some of them anyway. For example, Colonial Sugar Co., using a phosphatation process, were not interested in a filterability test and expressed some doubt as to the value of Amstar's ash standard for defining one important characteristic of raw sugar; that is, its effect on scaling of heating surfaces. Their conclusion was that, to provide a meaningful evaluation of the refining qualities of a raw cane sugar, the development of a standard affination procedure is necessary, taking into account the viscosity of the syrup adhering to the raw with appropriate testing of the washed sugar so obtained.

Supreme in Louisiana is both a raw sugar factory and a refinery, and is consequently well placed to be aware of the problems from both the producer's and the refiner's side. In addition to the raw from its own production, it refines the entire output of various other Louisiana mills. Following a falling-off in the refining quality of the sugars being produced, a meeting was held in 1965 with representatives from these mills and it was decided that there should be a set of raw sugar standards acceptable to both sides. Supreme was thus the first refinery in the U.S.A. to apply a set of standards and penalties in the buying of raw sugar. However, these were limited to sugar purchased from mills within the state of Louisiana, and premiums were not included.

Again, the viewpoint was taken that as the processing was different from that of other refineries so the problems would be different, and eventually

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standards were set for only three factors, temperature of sugars as received, invert content and grain size. It was the opinion of Supreme that, if these standards were met, the other factors involved would be acceptable with the exception of ash. Nevertheless, ash was not included in the standards because it was not a problem in ravs produced from Louisiana cane. The standards set were as follows:

Temperature: 38°C (100°F) or less
Invert: 0–1.00%
Grain size: 0–4.9% through 35 mesh Tyler screen

Temperature was included because it had been observed that sugar stored at a temperature initially in excess of 38°C (100°F) could double in colour in six months. A table was prepared listing the penalties deductable from the raw sugar price as the standards were exceeded by gradually increasing amounts, and the upper limits for each criterion were set as follows:

Temperature: 49°C (120°F)
Invert: 1.55%
Grain size: 14.9%

At these levels, each factor would carry the maximum deduction of 2% of the price; if any factor exceeded the maximum in the table then the sugars were rejectable and subject to negotiation. These standards were first applied to the 1966 crop and served very effectively for the short time they were in use.

The C & H refinery at Crockett in California had been early to start a joint programme of work with their raw suppliers in Hawaii to define and produce a sugar which would minimise refining costs. This led to the introduction of quality standards which were initially set as follows:

Pol: 97.5–98.8°
Grist: Affined sugar grain going through a 28 Tyler sieve not to exceed 34%
Filterability: 220 under standard Hawaiian test
Colour: 6 units (each unit being ten times the specific absorption at 420 nm)
Ash: 0.75% on unwashed sugar

A schedule of allowances was then drawn up which was based on previous refining experience which related costs to the quality of raw processed.

Other American refineries produced their own quality standards, though this did not of necessity mean that penalties and premiums were applied. However, it is likely that each refinery, not unnaturally having a slightly different process or different areas of difficulty, would tend to assess their raw sugar in different ways. Godchaux-Henderson, for example, calculate the analysis of the molasses that would be produced from the raw sugar under ideal conditions, and using this figure estimate the pounds of raw sugar which would be required to make 100 lb of refined sugar if the pol of the raw sugar were 96 and if the raw sugar were ideally refined with no resulting undetermined sucrose losses. This figure is called the ideal conversion; a standard conver-
sion figure is also produced which includes an undetermined sucrose loss of 0.62% and an elimination of 11% of incoming impurities before molasses production. Other criteria considered to be of importance are the invert, ash and organic/ash ratios, the composition of the ash and the grain size distribution.

However, most cane bought for refining in the United States is now purchased under the terms of the so-called No. 10 contract of the New York Coffee and Sugar Exchange; the sugars deliverable under this contract are defined as:

(1) Foreign growth cane sugars duty paid
(2) Duty-free cane sugars
(3) Cane sugars the product of continental U.S.

The standard quality specifications as defined by the No. 10 contract are based on the original Amstar standards and are as follows:

Factor of safety: Not exceeding 0.30
Ash: The maximum standard ash content is derived by multiplying percent non-sucrose solids by the factor listed, which corresponds to the final polarisation of the cargo.

<table>
<thead>
<tr>
<th>Polarisation (%)</th>
<th>Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Up to and including 98.0°</td>
<td>0.32</td>
</tr>
<tr>
<td>&gt; 98.0° to 98.2°</td>
<td>0.33</td>
</tr>
<tr>
<td>&gt; 98.2° to 98.4°</td>
<td>0.34</td>
</tr>
<tr>
<td>&gt; 98.4° to 98.6°</td>
<td>0.35</td>
</tr>
<tr>
<td>&gt; 98.6° to 98.8°</td>
<td>0.36</td>
</tr>
</tbody>
</table>

Grain size: through 28 mesh Tyler 20-55
Filterability: ml per 10 minutes 45-140
Colour: ICUMSA Method 2, 1958 100-230

No delivery of sugar is permitted testing below 94° pol.
The schedule of allowances and penalties under the contract are as follows:

1. Polarisation
   For the full degree 96.0° to and including 97.0° add 1.5% of the c.i.f. price.
   For the full degree 97.0° to and including 98.0° add an additional 1.25% of the c.i.f. price.
   For the full degree 98.0° to and including 99.0° add an additional 1.0% of the c.i.f. price.
   For the full degree 96.0° to and including 95.0° deduct 1.75% of the c.i.f. price.
   For the full degree 95.0° to and including 94.0° deduct an additional 2.75% of the c.i.f. price.

Fractions of a degree in proportion: any shipment polarising over 98.8° shall be considered for the purpose of polarisation allowance as testing 98.8°.

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2. **Moisture**
   Factor of safety: for each 0.01 in excess of 0.30 deduct 0.06% of basis price.

3. **Ash**
   For each 0.01% of ash content in excess of derived maximum standard ash content deduct 0.01% of basic price; for each 0.01% of ash content below derived minimum standard ash content add 0.005% of basis price.

4. **Grain size**
   For each 1% above 55% through Tyler 28 sieve deduct 0.04% of basis price, for each 1% below 20% add 0.02% of basis price.

5. **Filterability**
   No premiums or discounts.

6. **Colour**
   For each 1 unit above 230 deduct 0.01% of basis price, for each 1 unit below 100 add 0.005% of basis price.

It is laid down that testing of the samples be carried out by three laboratories, one acting for the seller, one for the buyer, which would normally be the buyer’s own refinery or central laboratory, and the third one being the New York Sugar Trade Laboratory. The contract does not define in words the methods of test to be used but merely states that they shall be those used in the trade for testing the whole raw sugar for moisture and ash and for testing affined raw sugar for grain size, filterability and colour. To determine the base figures for each quality specification, an average of the two nearest of the three test results is used. It is laid down that the producer and receiver of the sugar may waive any or all of the financial adjustments by mutual agreement, and, in fact, because of the difficulty involved in devising a reliable and reproducible filtration test, the filterability factor is now largely disregarded.

The Queensland government’s raw sugar quality scheme covers five factors: pol, starch, colour, grain and filterability, which are the subject of specification. So far as British refiners are concerned, they have been content to take what comes and do the best they can with it. This attitude was perhaps understandable in the past when raws of many different origins were refined and the possibility of getting meaningful specifications to cover the characteristics of all of them was remote. However, this attitude is now changing; Tate & Lyle is certainly moving towards the setting of acceptable quality standards for raw sugar. Several years ago a draft proposal was prepared for a specification for raw sugar which it was considered could be refined without undue additional expense. After stating that the sugar should be homogeneous and free from syrupy lumps, metal and other objects which could damage handling and processing plant, various analytical maximum limits were listed as follows:

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(1) Invert: less than 1.00%
(2) Ash: less than 0.60%
(3) Safety factor: less than 0.25
(4) Colour/ash ratio: less than 6000
    (colour measured by method 1A as listed in the proceedings of
     ICUMSA Session 12, 1958, p. 55)
(5) Starch: less than 200 p.p.m.
(6) Gums and waxes: less than 150 p.p.m.
(7) Yeasts: less than 10,000 live cells/ml 10% w/v solution
(8) Affined sugar size distribution:
    Mean aperture more than 0.020 in.
    Coefficient of variation less than 35%
    Gross conglomerates less than 20% of the particles
    Fines less than 3% through 60 mesh
(9) Ash in the affined sugar: less than 0.1%

These factors are more numerous than those in either the American or
Australian specifications, though whether they would all appear in any final list
is not yet known. We must be clear what the specifications are for. They are
primarily to indicate to the producer the quality of sugar required and to give
him some sense of urgency in achieving that quality by the imposition of price
penalties. If the producer just accepts the penalties and makes no effort to
improve quality then the exercise will have failed. The refiner is more
interested in having a steady supply of standard quality sugar which he can
refine with minimum difficulty than in purchasing less favourable raws at
bargain prices.

VI. COSTING

If a decision is made that raw sugar quality standards are to be set, then a
decision must also be made as to whether any contract of purchase will also
include penalty clauses or premium clauses, or both. In this respect, as has
been explained in the previous section, the penalty clauses are of more interest
to the refiner; he is not particularly interested in getting sugar of better quality
than he requires (indeed in some respects this may be a disadvantage), but
rather in getting the sugar of the quality he requires. It must then be decided
what factors are going to be included in the contract and what the level of
penalties (and/or premium) is going to be. Two difficulties arise; firstly, that it is
virtually impossible financially to assess the detrimental effects of some of the
factors which have been mentioned and certainly might play an increasing part
in the future standards. An example would be the level of trace elements or the
possible presence of traces of herbicides and insecticides. These factors are
bound to become of increasing importance as the users of white sugar set
higher and higher standards, but, so far as pesticide residues are concerned, we

have as yet not even got a satisfactory method of testing for them, nor in the main have legal limits been set. The second difficulty is that, for the producer supplying many different consumers, each of his customers might try to negotiate a contract for sugar rigidly tailored to his own needs in every aspect; splendid for him but presenting grave problems to the producer if every contract is different.

Other difficulties arise; for example, is a simple colour specification of any great value if the colour type or quality is not known? For some colours are very readily removed, while some persist right through into the refined sugar. It is only recently that some of the latter have been identified. It might be possible that at some time the producer will be able to control the level of these colours; perhaps that level might be specified in a contract, but that day has not arrived, nor is it likely to for some considerable time.

One other point to be considered is that whatever quality factors are included in a contract, it will normally be necessary for each to be measured by two or three separate analysts, one for the sellers, one for the buyers (which would normally be his own laboratory), and possibly by an independent third party as, for example, is currently the case for raw polarisation measurements where there is disagreement. For the other tests an average of each ship's cargo would probably be sufficient, but whatever tests were decided on, costs would be involved in carrying them out. If the tests were complicated and difficult these costs might be quite high; they would be higher still if the tests were such as not to be readily reproducible and many samples had to be sent to independent arbitrators. The result might even be that the cost of doing the tests would be greater than any benefit likely to be gained from penalty clauses, as it would probably only be in the minority of cases that these would be invoked.

These then are some (possibly not all) of the considerations to be borne in mind when deciding on what factors to include in any contract. Summarising, the criteria would appear to be:

1. The effect of the factor must be measurable.
2. It must be possible to translate the effects of the factors into financial terms.
3. The test used to measure the factor must be standard and reproducible.
4. The factor is preferably one which will apply to all refiners and not to an individual buyer only.
5. The factor must be one over which the producer has some control.
6. The costs of carrying out tests must not be excessive.
7. The choice of tests should bear in mind the relations between properties, such as between pol and ash, filterability and starch content, and moisture content and safety factor.

These criteria will apply some limit to what factors would be included in a contract; reconsideration of the list given in Section III might then yield the following as those which might be included:

(1) Polarisation
(2) Safety factor
(3) Ash
(4) Affinability (i.e. crystal characteristics)
(5) Filterability
(6) (possibly) Colour

It would be necessary to list the limits of these factors, and the problem then arises as to how the level of any possible penalty (or premium) is to be fixed.

There would appear to be two approaches to this problem. Though neither can give an accurate result, possibly they can give a basis for putting down initial figures which could be modified in the light of experience. The first is to take out the refining costs over a period of time and try to relate these to the quality of the raw sugar being worked. This period of time would probably have to be at least a year, during which all the factors would have to be measured and an attempt made to assess their individual effects on refinery costs. For a refinery dealing with a fairly constant raw this method is hardly practicable, as the various factors would not change in level sufficiently for the effects of the change to be noticed. It might be more practicable for a refinery dealing with a variety of raw sugars, except that there cannot be a clean break between one delivery and the next. The effects of one are carried over into the next, and indeed, a refinery can very easily be unloading and taking into the refinery sugar from two ships of different origins at the same time. All these difficulties must be realised when using this method to assess costs. Nevertheless, it has been used by some refineries for setting the level of cash penalties.

The second method is to try to assess the level of the effect of each factor going markedly outside specification and apply to this a costing (which would be based on normal refinery costs). This method has the advantage that it can be assessed in isolation from the refinery, though of course, it would be based on refinery data; it has the disadvantage that initially the figures would be based on various assumptions. However, whatever method is used, some figures can be produced which can be used initially; later experience will no doubt refine them to something more satisfactory.

It is not proposed to discuss the first method in any detail: it assumes that all refining costs are accurately known for each unit process. However, it must be realised that the effect of one factor can be felt in more than one part of the process, and indeed might put the costs up elsewhere in the refinery more than in the department where the difficulties of this factor are actually being experienced. Take a badly filtering raw, for example. In the filtration department this may lead to slow filtration and hence to loss of melting rate. If there is spare filtration capacity or if the loss of melting rate is not at that time vitally important, it may be possible just to carry on until the bad patch is through. In the first case, with spare filtration capacity available, then there might be some increase in labour costs in order to put more filtration capacity into use; if it is
decided to reduce the melting rate, it is possible to correlate this reduced rate against normal costs in the filtration department. However, it may be that the refiner has no spare capacity, nor is it possible for him to reduce his rate of melting. In this case filtration must proceed at the normal rate involving the use of more filter aid or more lime in a carbonation plant, more frequent cleaning of presses, more frequent renewal of filter cloths and almost certainly the use of water to reduce the solids content of the sugar liquor to a level that will enable it to go through the filters at the required rate. Thus, in the filtration department, costs will be involved for filter aid, cloths, and labour, but it is suggested that the total costs of these might be outweighed by the cost of evaporating the extra water added. Thus, the principal cost of a poor filtering raw may be found in the steam-raising plant. Similarly with a badly affining raw, this might involve extra costs at the affination station but this is unlikely. The principal costs will be involved in recovering the extra sucrose washed into the recovery plant and possibly dealing with the effects of a poorer quality melter liquor, which will certainly be felt throughout the whole refinery. These instances are quoted to indicate the difficulties inherent in using departmental costs as a means of fixing the level of penalties, or of allocating total refining costs amongst the various factors. It is not suggested that these difficulties cannot or have not been overcome; indeed, it has been reported in at least one instance that this method has been used to fix the level of penalties.

The second method involves a direct calculation of the extra likely costs involved for each factor (it is being assumed, initially at any rate, that premiums are not involved). Again it might be necessary to use previously acquired information, but this time analytical rather than financial. Thus, hopefully, it is possible to assess the physical effects of each factor going outside specification and correlate this with the level of each contract factor. Thus, using filtration as an example again, a raw outside specification to a certain extent may add 2% to the water content of the sugar liquor being filtered, a sugar of very low purity may lead to excess sucrose going out in the molasses to the level of (say) 1%; it will certainly lead to more strikes of all crops being boiled. Poorly affining raws will mean more sucrose into the recovery house, poorer decolorisation and more wash (of which a record will be kept) on the white sugar machines.

The factors previously mentioned which do, or might, play a part in any contract for the purchase of raw sugar are now considered separately and in detail.

A. Polarisation

Raw sugar is normally purchased on the basis of 96° pol, and in the United Kingdom this is the only factor for which there is, and has been for a considerable time, a financial adjustment of the purchase price according to the level of polarisation at a rate agreed between buyer and seller and which will

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probably remain at this rate. The terms of the contract are:

(1) For each degree above 96.0° add 1.4% of the c.i.f. price. The premium is not payable for sugars over 99° pol if they come from Commonwealth sources, or for foreign sugar over 98° pol.
(2) For sugars of 96.0–95.0° deduct 1.5% of c.i.f. price.
(3) For sugars of 95.0–94.0° deduct an extra 2.0% of c.i.f. price.
(4) For sugars of 94.0–93° deduct an extra 2.0% of c.i.f. price.

Sugars under 93.0° pol do not come within the terms of the contract; all part degrees are paid for pro rata.

For sugars from Central American sources a slightly different contract might be used; for premium this is the same as the American No. 10 contract but for penalties it is slightly different.

(5) For sugars 96.0–95.0° deduct 1.6% of c.i.f. price.
(6) For sugars 95.0–94.0° deduct an extra 2.0% of c.i.f. price.
(7) For sugars 94.0–93° deduct an extra 2.5% of c.i.f. price.

It is, however, very unusual for any sugars supplied to the United Kingdom market to bear a penalty; in the great majority of cases the sugars achieve a substantial premium by being well over the 96.0° lower limit.

B. Safety factor

This is an important factor and would be fixed at a level which is both satisfactory to the buyer and easily achievable by the producer. The Tate & Lyle suggested limit is 0.25; the American No. 10 contract quotes a figure of 0.30. Unfortunately, or rather perhaps fortunately from the refiner's viewpoint, there is little published information on this factor and it is not one on which the refiners themselves care to gain experience, though as previously quoted Tate & Lyle have been involved in one case of marked deterioration. Following this and an extensive survey of Mauritius sugars by the Mauritius Sugar Industry Research Institute®, figures were published which could be used as a basis for calculating likely losses. However, this is a factor which is unlikely to be outside safety limits, though clearly the possibility must be appreciated.

It will be noticed that temperature has not been indicated as a factor to be included in a contract of purchase. This is because it has no relevance for the buyer of off-shore sugar, which makes up the whole of the U.K. market and a large part of the American market, though for purchases of sugar of domestic production, such as Louisiana and Florida raws for the U.S. Southern refineries, such a factor might have relevance. Temperature of input into the raw sugar terminals is the important figure; any damage that is going to be done by storage at too high a temperature will have been done by the time the sugar reaches its final market.

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C. Ash

It is generally assumed that an acceptable maximum level for the total ash content of affined sugar is 0.10%. If a further assumption is made that affination removes 85% of the ash from the whole sugar, then this would fix the maximum desirable ash in the raw sugar at 0.67%. In practice, a figure of 0.60% might be considered as a standard. The effect of a higher ash level could be threefold: it could increase the ash in the affined sugar, it could lead to more sucrose being washed into the recovery house, and it will certainly mean more sucrose going out of the process in the form of molasses.

Concerning the first factor, the increased ash in the affined sugar in its turn may lead to an increased ash content in the treated sugar liquor for decolorisation, and there is strong evidence that the higher this ash content is, then the poorer is the decolorisation by bone char. The ash content of the decolorised liquor will also be higher, and these two factors will or might lead to the use of increased wash water on the white sugar machines and increased evaporation costs. Unfortunately, it does not seem possible accurately to assess the financial costs of these effects, but they must be considered when fixing any penalty for excessive ash content in the raw sugar.

So far as the second effect is concerned, i.e. the increased load on the recovery house, then previous experience will prove a good guide. It is also possible to assume that it will require the application of a certain amount of wash water above the normal to achieve the desired results at the affination station (again previous experience may be a guide), and to calculate the additional costs involved in recovering the extra sucrose.

The cost of the third effect, that of the increased molasses, can be fairly readily calculated. Take, for example, two raw sugars, one at the top end of the allowable ash scale, the other well above it.

<table>
<thead>
<tr>
<th></th>
<th>Sugar 1</th>
<th>Sugar 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>%Pol</td>
<td>97.30</td>
<td>95.78</td>
</tr>
<tr>
<td>% Invert</td>
<td>0.95</td>
<td>1.57</td>
</tr>
<tr>
<td>% Ash</td>
<td>0.60</td>
<td>1.00</td>
</tr>
<tr>
<td>% Water</td>
<td>0.40</td>
<td>0.40</td>
</tr>
<tr>
<td>% Organic</td>
<td>0.75</td>
<td>1.25</td>
</tr>
</tbody>
</table>

The ideal molasses obtainable from these can be calculated from the 1962 Tate and Lyle formula:

\[ Z = \frac{100(5 + 3g)}{3(3 + g)} \]

where \( g = \% \text{ invert}/\% \text{ non-sugars} \).

In the two sugars quoted above, \( g = 0.7 \), so \( Z \), which is the \% total sugars on a dry basis obtainable from them, would be 63.96. This, of course, is the ideal molasses, not the actual that would be obtained. In practice, the total
sugars obtained would probably be higher because no refinery runs in an ideal manner.

Thus the analysis of the molasses obtained from the two sugars would be the same; what would be different is the amount produced. Thus, if 100 tons of the first sugar is taken, the amount of molasses (on a dry basis) which would be obtained if it were refined ideally is given by the formula:

$$\frac{100 \times \text{weight of non-sugars in material}}{\% \text{ non-sugars in molasses}}$$

$$= \frac{100 \times 1.35}{36.04}$$

$$= 3.75 \text{ tons, or on a wet basis assuming}$$

$$25\% \text{ water in the molasses 5.0 tons.}$$

Similarly from the second sugar, there would be obtained

$$\frac{100 \times 2.25}{36.04}$$

$$= 6.23 \text{ tons or 8.32 tons wet molasses.}$$

Thus:

<table>
<thead>
<tr>
<th></th>
<th>Sugar 1</th>
<th>Sugar 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tons actual raw</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Tons dry material (a)</td>
<td>99.6</td>
<td>99.6</td>
</tr>
<tr>
<td>Tons white obtained (a – b)</td>
<td>95.85</td>
<td>93.37</td>
</tr>
<tr>
<td>Tons dry molasses obtained (b)</td>
<td>3.75</td>
<td>6.23</td>
</tr>
<tr>
<td>Tons wet molasses obtained</td>
<td>5.00</td>
<td>8.32</td>
</tr>
</tbody>
</table>

These figures can then be used to calculate the loss in money return due to there being an excess of ash in the raw sugar input. However, it must be appreciated that, for the calculations shown above, it has been assumed that the only output from the refinery is white sugar and molasses. Though the basis of the calculations would remain the same, some adjustments might be necessary if part or all of the raw sugar input was used in the production of by-products.

D. Affinability

The effects of bad affinability of raw sugar are in practice similar to those due to high ash content, that is, that more wash water will be required on the affination machines to produce the same quality affined sugar. This will mean extra costs being involved in recovering the extra sucrose washed into the recovery process, but there will, of course, not be any additional molasses made because of this. Previous experience of the likely wash increases will be the best guide on which to base calculations, and if it is not possible, even with

extra wash, to obtain the quality required, then, as in the previous section, there will be extra costs involved in reduction of decolorising capacity and in the use of extra wash water on the white sugar machines.

E. Filterability

Various factors cause extra costs if the filterability of a raw sugar is poor, but it is likely that the most important consideration will be the cost of evaporating the extra water which will be added to get the sugar liquor through the filters at the required rate. The problem is, of course, knowing how much extra water will be added. Previous experience will be a guide, or it might initially be considered best to make some assumptions.

If the normal water content of the filtered liquor is, say, 33%, then for every 100 tons of sugar in the filtered liquor there will be used 49.3 tons of water. If the refinery has a recovery house, and the only products are white sugar and molasses, then the recovered sugar will normally be returned in some form to the main stream and the 100 tons solids will contain both affined sugar and recovered sugar. The only input into the refinery that would not be filtered would be (say) 5% that would go out as molasses. Thus the 49.3 tons of water would be the equivalent of

\[
\frac{100 \times 100}{95} = 105.3 \text{ tons of raw sugar}
\]

If it proved necessary to increase the filtered liquor water content to 35% in order to process 105.3 tons of raw sugar in the same time, then the water used would be

\[
\frac{100 \times 35}{65} = 53.9 \text{ tons}
\]

Thus for every 105.3 tons of sugar processed, an extra 4.6 tons of water need to be evaporated. The cost of doing this can be calculated on the basis of the normal refinery evaporating costs. At Liverpool refinery, for example, the direct evaporation costs for one year at normal throughput rate would increase by £60,000 if it were necessary to filter the whole throughput at 2% more water content than normal. The word “direct” is stressed because there is (as always) a complication in that part of the evaporation may be done in a multiple effect evaporator, possibly using heat that might otherwise be wasted. However, these complications can be taken into account in the calculations.

The other direct extra costs involved in poor filtering raws would be any labour costs involved in putting extra filtration capacity into use, the cost of extra filter aid (including lime on carbonatation plants) and the cost of possibly renewing press cloths more frequently. There is unlikely to be any great difficulty involved in making suitable allowances for these factors.

The effect of the level of the colour in the whole raw sugar tends to be rather unpredictable; what is more important is the type of colour which is present. However, as there are no means available at the moment for separating quantitatively those colours which persist through into the final product, we have to be content with fixing a figure for the sum of all the colouring materials present. This will probably be fixed initially at a level judged suitable by past experience. For calculating any penalties an assessment must be made of the likely effects of the higher colour which will be apparent even after the decolorising process and which will lead to increased wash on the white sugar machines.

G. General

Two methods have been discussed for calculating and fixing the penalties and premiums which might be embodied in a contract. There is, of course, a third method: that of getting by agreement some figures down on paper, seeing how they work out in practice and adjusting them in the light of experience. However, whatever method it is decided to use, it must be remembered that the penalty clauses must be such as to make it worthwhile for the producer not to exceed the agreed limits, and also that the user hopes that it will never be necessary to invoke them. Evidence published so far of the use of quality standards shows that the vast majority of raw sugar falls within them.

VII. THE ROLE OF THE RAW CANE SUGAR PRODUCER

It has been observed in a previous section that any factor built into a contract must be one over which the producer has some control. This does not mean that every factor over which he has control will be included. For example, the grower and miller have control over the type and amount of any pesticides, herbicides and bactericides used but, as there are no readily available means of detecting or measuring residues of these materials, it is at the moment impossible to specify any limits for raw sugar. In this respect the user is completely dependent on the producer to ensure that the materials which are used are satisfactory for the purpose, and of such a nature that any traces left in the raw will be removed during refining. However, as the demands from white sugar users become more critical so will the demands made on the producers by the refiners become more critical; it is probable that the limiting factors included in contracts will be extended beyond the half-dozen or so already in use, and the most likely to be considered for inclusion first are limits for trace constituents including elements as well as herbicides, etc.

Even before the introduction of quality standards, much had been done over many years to improve the basic qualities of the raw sugar being supplied.
by the producers to the refineries. There has been, for example, a general improvement in sugar quality, with the result that there are not the wide variations between sugar from one origin and another that were liable to occur years ago, a change that has eased refining problems in the United Kingdom. As a result of this, by far the greater part of sugar produced now falls within acceptable levels and the evidence is that, where they are in being, penalty clauses are rarely invoked.

All the factors as previously listed in Section VI, including temperature of the raw sugar, can be completely or to some extent controlled by the producer. Even dextran, which until fairly recently had very little attention paid to it, can now be dealt with, although its quantitative estimation is difficult owing to its rapid growth in uncrushed cut cane. To minimise dextran increase, the maximum time between cutting and grinding should only be a few hours. Furthermore, a recent paper describes work on the removal of dextran from deteriorated cane juice by the use of dextranase. The authors describe their work and recommend a process but add that the process is not recommended for general use, except under conditions of breakdown in the mill or unavoidable hold-ups in cane supply. Apart from its cost, the general use of the process would conceal inefficiency; the problem of cane deterioration should be overcome by correct measures being taken in the fields. The authors of this review absolutely agree with this viewpoint, not only with regard to the circumscribed field of cane cutting, but with every aspect of the production and refining of sugar. No-one should be engaged in putting right things that should never have gone wrong; unfortunately too many people are.

The level of polarisation and the level of ash are interdependent. Though raw sugar is bought on the basis of 96.0° pol, this level was fixed commercially, and most of the sugar purchased, certainly within the United Kingdom and American markets, is of much higher purity than this. If it were not so, the maximum level of ash content considered as a possible standard for the United Kingdom market would often be exceeded.

However, producers supplying sugar to a world market are likely to have to supply to a variety of quality standards. This problem has been tackled in South Africa by the initial production of nothing but a single very high quality sugar. This is effected by double curing and remelting of “C” sugars and by the use of “B” sugar to make magma for footings for “A” massecuite. Consequently, nothing but “A” sugar is produced and this is further purified by steam-washing the crystals in the centrifugal machines. The very high purity sugar produced is stored until it is required for delivery, when it is coated to the required purity with high test molasses produced by inverting syrup from “A” massecuite until 60% of the sucrose is inverted and then concentrating to 82% solids. Alemán describes a somewhat similar method of producing sugars of satisfactory quality, in this case two sugars of virtually similar properties but not steam washed in the baskets. He adds that the massecuites must be cooled off before machining and the purged sugar needs to be aerated so that it reaches the bulk storage at a temperature no higher than 38°C (100°F).

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So far as the grain quality is concerned, the methods of full seeding as described produce not only the best quality sugar but also the best quality grain. There are three methods available for inducing grain into a masscuite: graining, shocking and seeding. In the first, evaporation of the charge is carried on until crystal formation starts. When the pan boiler judges that sufficient crystals are present, he stops any more forming by reducing the degree of supersaturation, either by adding a lower brix syrup or by increasing the temperature. The method of shocking involves the evaporation of the pan charge to a proper degree of supersaturation, at which point crystallisation is induced by shocking with a small amount of sugar dust. In this method, also, the pan boiler has to judge when sufficient crystal nuclei are present and then take measures to prevent any more forming. Both these systems are difficult to control satisfactorily, the first particularly so, requiring, as it does, a very high degree of skill by pansmen. The number of crystals formed has to be just right; too many means that the final sugar will be too small with consequent difficulties in purging, too few will make the main grain of the sugar too large and in this lies grave danger in the boiling stage. If there are too few crystals present or those that are present are too large, then the surface area available for the crystallisation of sucrose is less than it should be. Thus there is always a tendency, if the evaporation rate is high, for fresh crystal nuclei to form and these will appear in the final sugar as very small grain. The result will be a large main grain with a secondary crop of small crystals, which is an ideal combination for bad purging in the affination process. The system of full seeding is now becoming fairly generally adopted. It involves the addition of the correct number of crystal nuclei, using either a magma made from the smaller crystals obtained from a lower grade masscuite as in the cases already noted, or, if it is being used for white sugar boiling, using a slurry prepared from finely milled white sugar. The method, in theory, should guarantee that the main grain of the final sugar will be the right size. However, the production of the perfect crystal depends on more than the method of seeding. For example, if the pan circulation is bad, there will be a tendency for crystal conglomeration to take place. Good pan circulation, either by effective design or induced circulation by means of a stirrer, is essential if the best crystal is to be obtained.

Having obtained a satisfactory crystal, nothing should be done to spoil the good work. The introduction of continuous centrifugal machines has been a mixed blessing, splendid as a production unit but very damaging so far as sugar crystals are concerned. There seems to be little doubt that if crystal damage is to be minimised, batch machines would be the first choice. However, the production advantages of the continuous machine are such that, if they can be operated so that crystal damage is only slight, then such slight damage might be tolerable. Fortunately, the problem of crystal breakage with the first generation of continuous machines was very quickly recognised as serious, and the more recent machines are being produced with extra width monitor casings so that crystal breakage is greatly reduced if not completely eliminated.

The properties of raw sugar that have the major effect on its filterability

are, even now, not clearly defined. However, whether one believes that starch is a major contributory factor or not, it is certainly true that its absence from raw sugar, or, at least, its presence in only small amounts, will do no harm and might even do some good. Various processes have been proposed and tried to control the starch content of raw sugar. Amongst these has been the use of a centrifuge, proposed by South African workers. Unfortunately, it was found that to reduce the starch level significantly, the rate of throughput had to be very much lower than was necessary. Vacuum clarification, the so-called Rabe process, has been tried in South Africa. In this process, lime is added to the raw juice to adjust the pH to a level between 7 and 9. The presence of orthophosphoric acid or monocalcium phosphate is to coagulate the impurities, including the starch and gums, and the coagulated materials are brought to the surface of the juice by subjecting it to reduced pressure. They are removed, and the purified juice sent forward for further treatment. However, it was found that control of the vacuum flotation was very sensitive to variations in both juice flow and juice quality and the process was abandoned.

Various methods have been proposed for making use of enzymes to reduce the starch content of sugars. In 1926, Haddon used a heat-stable enzyme for this purpose. It appears to have been the first use of such a method which, although patented, appears to have received little attention. So far as South Africa is concerned, not much was done in this field until the appearance of bacterial amylases at competitive prices. Most factories in South Africa now use these materials normally, adding them to the juice between the last two vessels of the evaporator. As the enzymes are virtually free from invertase, they can be used for starch hydrolysis without danger of any associated sucrose loss. Another method, using the naturally occurring enzymes present in cane juice, is the subject of an Australian patent and was investigated by Nicholson and Horsley of the Colonial Sugar Refining Co. The method depends on the fact that the enzymes already present in the juice will hydrolyse the starch, provided the conditions are right. The juice is held for a short period at a pH not far removed from that of natural juice and at a temperature high enough to gelatinise the starch but not high enough to destroy the enzyme rapidly. Two methods can be used: one consisting of heating part of the juice to gelatinise the starch and, after cooling, mixing it with unheated juice still containing active enzyme, the other consisting of heating all the juice to a temperature near 70°C and holding for a period. Juice must not be limed before heating.

The method of juice extraction from the cane can also have an effect on the starch content of the sugars produced. For example, it has been suggested that diffusion introduces less starch than crushing, and if crushing is the process followed, it is desirable to use cold rather than hot water, at least so far as waxes are concerned (and in dealing with methods to improve filtration these might be classed with starches).

Finally, we come to the question of colour. With those naturally occurring enzymes.
in the juice, the grower has no control except perhaps by growing a different variety of cane. Colour can be very rapidly developed by overheating in any part of the process. It has also been suggested that much colour generation (and also sucrose loss) can take place if the juice is overlimed during the clarification stage. To achieve minimum colour generation in the raw sugar factory, the same principles must be followed as those that apply in a sugar refinery, that is to work as cool as possible and to minimise retention times.

One other factor which can affect perhaps not the colour level but certainly the ease with which it can be removed in the refining affination process, is the quality of the crystal. Badly conglomerated grain will make all impurities, including colour, more difficult to remove. Furthermore, imperfect crystallisation may lead to the inclusion of an excessively large amount of colour within the crystal. Thus, two raw sugars might have the same colour but they would behave very differently in refinery practice.

Finally, it is essential that if the sugar is to be stored it must be cool enough to store without any danger of excessive colour formation. It appears to be generally accepted that, if this is to be so, the temperature going into store must not be higher than 38°C (100°F).

VIII. METHODS OF TEST

Because of the need to satisfy both buyer and seller, all analytical tests on the raw sugar will be carried out in two, possibly in three, laboratories. It is essential, therefore, that the methods of analysis employed be defined closely and also be such as to give reproducible results. No difficulty arises so far as the normal analytical procedures are concerned, viz, pol, ash and water, but difficulties arise with the other quality factors and particularly with the measurement of filtration rates.

A. Polarisation

Reference may be made to the two standard works on sugar analysis, ICUMSA Methods of Sugar Analysis and Analytical Methods Used in Sugar Refining. For information concerning the most recent discussions on polarisation techniques, reference may be made to the Proceedings of the 14th and 15th Sessions of ICUMSA. These, however, in no way alter the general principles laid down in the two former volumes.

B. Ash

The same considerations apply as for polarisation. The methods are clearly defined by ICUMSA and can be found by reference to the appropriate standard works.

C. Water

As for polarisation and ash.

D. Filtration

Many filtration tests have been proposed. Apart from the fact that no method has yet been devised which is satisfactory for relating laboratory filtration tests to refinery performance, the tests themselves suffer from lack of reproducibility, so much so that, in many instances, filtration specifications have been abandoned as part of the quality standards for raw sugar, or, at least, if they are still part of the specification, the tests are not carried out.

However, in recent years, there is probably no single quality factor which has received more attention, or on which greater effort has been expended in an attempt to achieve satisfaction, seemingly so far in vain. Notes on some of the tests suggested follow.

1. Elliott test

This is probably the earliest filtration test devised, dating back to about 1923. The apparatus consists of a standard size container fitted with a circular filter leaf of 4 in. diam. filtering surface, which is covered with an 8-mesh wire screen and then a standard, closely defined, filter cloth. Also required is a receiver of 4 litres capacity capable of withstanding full vacuum, together with the necessary vacuum tubing and gauges. Two thousand grams of sugar are dissolved in an equal weight of water and 40 g of standard filter aid added. The sample is poured into the filter vessel and subjected to a vacuum of 660 mm (26 in.) for exactly 30 minutes. The suggested standard filtration temperature is 27.5°C and the time to filter 3000 g of filtrate is taken as standard. Then the Elliott filtration rate is

\[
\text{Weight (g) filtered in 30 minutes} \times 100
\]

\[
\frac{3000}{\text{Weight (g) filtered in 30 minutes}}
\]

Like all filtration tests, to have any chance of reproducible results the details of the test must be strictly adhered to. Many modifications have been suggested and one may conclude from this that no completely satisfactory procedure has yet been worked out.

2. Bomb filter

This filtration test was developed by the technical staff of the Johns Manville research laboratories. The bomb consists of a pressure vessel 11 in. deep and 5 in. i.d. narrowing to 3 in. at the bottom. The filter leaf is 1½ in. diam. fitted with a standard filter cloth. Agitation of the contents of the bomb can be carried out either by air or by means of mechanical stirrers. The tests are carried out at 60% solids solutions for raw sugar and 65% solids for affined sugar.

sugars, and a precoat of standard filter aid is used. The test differs from the Elliott in being operated at a temperature of 80°C, which is maintained by the use of a thermostatically controlled oil bath. Tests are run on a definite pressure increase schedule which can be controlled either automatically or by hand, and filtration volumes are read at definite time intervals. The test has gained some favour, but like all filtration tests requires rigid adherence to standard conditions to achieve reproducible results.

3. Colonial Sugar Refining Co. test

This test was developed by Nicholson and Horsley in the C.S.R. research laboratories, and was designed to fulfil their need for a filtration test which could be operated with only small quantities of sugar. Because it was desired to use the test in the company’s mills and refineries as well as in the research laboratories, it had to be simple, portable and independent of services such as steam, electricity and air pressure lines. In the event, the filter consisted of a piece of brass tubing of 2 in. i.d. and about 9 in. long, the base of which was threaded and designed to hold the filter disc, which was a circular brass plate drilled with 45 $\frac{1}{2}$ in. holes and over which was fixed a piece of 120 mesh Monel gauze, edge-soldered in such a way that the solder could not creep onto the surface of the gauze and so affect the filtering area. The head of the filter carried a gauge, pressure-relief valve and a threaded gas inlet connection. For test purposes, the filter septum was a disc of Whatman No. 1 filter paper (though later a change to Whatman No. 54 was recommended). As the test was designed to correlate refinery and laboratory test results, it was decided to operate at a pH of 9.0 and the solution was adjusted to this by the addition of a lime-sucrose mixture. As a buffer, triethanolamine and calcium acetate in 50% glycerol was used.

The standard conditions for determining the filterability of raw sugar were fixed as follows:

<table>
<thead>
<tr>
<th></th>
<th>60% solids ± 0.1%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration</td>
<td>9.0 ± 0.1</td>
</tr>
<tr>
<td>pH</td>
<td>0.5% on solids</td>
</tr>
<tr>
<td>Weight of standard filter aid</td>
<td>Room</td>
</tr>
<tr>
<td>Temperature</td>
<td>Constant at 50 p.s.i.</td>
</tr>
<tr>
<td>Pressure</td>
<td>8 minutes, the first 2 minutes’ filtrate being discarded and the next 6 minutes’ being collected.</td>
</tr>
</tbody>
</table>

The filter disc is previously calibrated by determining the rate of filtration of a 60% solids pure sugar solution using the standard quantity of filter aid, the rate being determined over a temperature range likely to be encountered under laboratory conditions. When a test has been completed, the filterability of the raw sugar is obtained by expressing the volume of solution collected in 6 minutes as a percentage of the volume of pure sugar solution collected at the
same temperature (which can be read off from previously prepared calibration graphs).

4. **Tate & Lyle pressure filter**

This is a bomb type pressure filter operated at an air pressure of 50 p.s.i.\(^{106}\). The internal stirrer is driven by magnetic induction from outside the vessel. Tests have been carried out using filter cloths of cotton, 350 mesh stainless steel and polyvinylidene chloride, with or without filter aid and either hot or at room temperature. If was found that reasonably reproducible results could be obtained when testing hot carbonatated liquor, but no correlation could be achieved between these and filtration tests carried out on the raws from which they were obtained.

This particular design originated at the Plaistow Wharf refinery of Tate & Lyle. Bennett\(^{106}\) has described a filter and method of test, developed in the same Company’s research laboratory, during the course of an investigation into the optimisation of carbonatation practices.

5. **California and Hawaiian Sugar Company**

The Elliott test is time-consuming, requires a large sample and has poor reproducibility. This alternative method is designed to give quick, reproducible tests with a small amount of washed sugar liquor. It consists of a simple constant pressure filtration of a 50% w/w sugar solution containing 1.0% filter aid by weight. The pressure filter is of C. & H. design, straight-sided and cylindrical, fitted at the base with a filter pad covered with a backing screen of a total filtering area of 4.33 in\(^2\). The filter paper used is quoted as National Filter Media Corp Alpha cellulose paper type AC 3101 and the pressure is applied by air at 20 p.s.i. To carry out a test, 170 g of the sample to be tested are dissolved in an equal weight of distilled water at 20°C and maintained at this temperature. Filter aid (1.7 g) is then added and mixed, the sample poured into the filter and the pressure raised to 20 p.s.i. Filtration is started exactly 1 minute after pouring the prepared sample into the filter. The filtrate is divided into two portions, that collected in the first half-minute, and that collected in the next 4 minutes. The total weight collected in 5 minutes is reported as the filterability; if colour measurements are to be made the filtrate from the first half-minute is discarded. A temperature correction can be made by subtracting 3% of the weight of the filtrate for each degree over 20°C and adding 3% of the weight of the filtrate for each degree under 20°C.

Advantages claimed for this design are that a small filter with a relatively large filtering surface gives a short filtration time, and thus dispenses with the need for stirrers and temperature control, and that the internal filtrate volume is held to a minimum by making the delivery tube both small and short. Essentially all the filtrate produced is measured.

*Sugar Technol. Rev.*, 3 (1975) 69–126
6. Amstar Corporation test

This method is based on work originally reported by Coll et al.\textsuperscript{107} of the Southern Regional Research Laboratory, New Orleans. It consists of the filtration of a 15% solids affined raw sugar solution maintained at 25°C through a 1.2 μm millipore filter at 22 in. vacuum for 10 minutes. The volume of the filtrate is adjusted by a factor which accounts for variations in the filtering characteristics of the membranes being used. Coll and his co-workers concluded that the millipore filter is sensitive to changes in raw sugar quality, is reproducible within 5%, apparently predicts refinery performance and is both quick and simple to operate. It also has the great advantage that the equipment can be bought off the shelf. Pearson and Ament\textsuperscript{108}, reporting on work carried out by the Amstar Corporation, concluded that tests carried out on laboratory washed raw sugar and refinery sugar liquor showed excellent correlation with refinery Sweetland press throughputs.

7. Plugging value test

This test was devised by Faviell and Blankenbach\textsuperscript{7} in the laboratories of the British Columbia Sugar Refining Co. The apparatus used consisted of a circular filter unit of exactly 2 in\textsuperscript{2} of filter screen area, the filtering surface itself being a piece of standard cotton press cloth. This was fitted to the base of a conical funnel and the outlet led into a graduated receiver to which vacuum could be applied. A stirrer was fitted so that the contents of the filter funnel could be kept mixed. The filter cloth was pre-coated and a 50% solids solution of raw sugar at a temperature of 20°C and containing 600 g raw sugar was poured into the top funnel reservoir. The stirrer was started and vacuum (15 in. Hg) applied. A stop-clock was started at the appearance of the first filtrate and the cumulative volume was then recorded against time until filtration stopped completely (in practice it was found convenient to plot the graph of volume against time whilst filtration was in progress, so that filtration could be stopped when sufficient points had been obtained to give a satisfactory straight line). It was reported that the plugging value test was found to be sufficiently sensitive to detect very small changes in raw sugar and filter aid quality.

8. Conclusion

From the number and variety of filtration tests described in the preceding pages (which are by no means all that have been reported in the technical literature), it will be clear that no complete satisfactory filtration test has yet been devised. The two principal difficulties are a lack of reproducibility and a difficulty in relating the results obtained in a laboratory test to refinery performance. These difficulties are such that filtration tests are being discarded from raw sugar quality standards—the American No. 10 contract contains no penalties or premiums for bad or good filterability. It seems to be another example of the difficulty of relating what happens in the laboratory to what happens on the plant, and so far as the tests themselves are concerned, unless they are reproducible, they can be considered only of limited value.

E. Factors affecting filtration

1. Dextran

The method normally used for the determination of dextran is based on the original work of Nicholson and Horsley as modified by Keniry et al. It depends on the measurement of the haze developed in 50% aqueous ethanol after the enzymatic removal of solubilised starch by the use of bacterial α-amylase, removal of calcium sulphate and other inorganic salts with ion exchange resins, de-proteination by the addition of trichloracetic acid and filtration. For the test, a 40% w/v solution of raw sugar is used. After removal of starch, inorganic salts and proteins and vacuum filtration on a suitable filter (Whatman No. 5), using a filter aid, 12.5 ml ethanol is added to 12.5 ml of the clear filtrate. The haze is allowed to develop for 20 minutes and read as absorbance at 720 nm in a 1 cm cell, using a suitable spectrophotometer. It should be noted that great care must be taken in the addition of the ethanol to the treated sugar solution. The alcohol must be added dropwise with constant swirling in order to avoid local alcohol concentration which may precipitate irreversibly substances other than dextran. When all has been added, mixing must be completed by gentle but not vigorous agitation. The amount of dextran in the raw sugar is then determined by reference to standard curves which have previously been prepared using commercial dextran in pure sucrose solution under identical conditions.

2. Starch

Numerous methods for estimating the starch content of raw sugars exist today, but most are based on the original method of Balch. The method depends upon the measurement of the intensity of colour produced by a starch–iodine complex; however, like many other analytical methods it has been modified to suit local conditions or in an attempt to improve it. There is not one agreed method and ICUMSA, the final arbiter on analytical methods concerning sugar, has only recently gone so far as to accept one variation as a tentative method, which means that it will not be accepted as an official standard method for at least four years.

Three methods will be described here: the South African Sugar Milling Research Institute method, the Tate & Lyle method and the Australian method devised by the Colonial Sugar Refining Co. It is this latter which has achieved tentative status within ICUMSA.

(a) S.M.R.I. Method. The reagents required are ethanol, acid-washed white kieselguhr filter aid, 2 N acetic acid solution, M/600 potassium iodate solution (0.3567 g/litre of previously dried reagent), freshly prepared 10% potassium iodide solution, 80% alcohol prepared by the dilution of absolute alcohol with water, pure potato starch, and 40% w/v calcium chloride solution adjusted with acetic acid to pH 3. A standard curve is first plotted by the following method. A solution of starch is prepared by adding 500 mg of dry starch to about 10 ml of water to make a slurry. This is added to 300 ml of

boiling water and boiling is continued for 1 minute. After cooling, the solution is transferred to a 500 ml flask and adjusted to volume: 1 ml, therefore, contains 1 mg of starch. The following solutions are then prepared:

<table>
<thead>
<tr>
<th>No.</th>
<th>Refined sugar, g</th>
<th>Water, ml</th>
<th>Starch soln., ml</th>
<th>Final starch concentration at colour level, mg/50 ml</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25</td>
<td>30</td>
<td>0</td>
<td>0.0</td>
</tr>
<tr>
<td>2</td>
<td>25</td>
<td>25</td>
<td>5</td>
<td>0.5</td>
</tr>
<tr>
<td>3</td>
<td>25</td>
<td>20</td>
<td>10</td>
<td>1.0</td>
</tr>
<tr>
<td>4</td>
<td>25</td>
<td>15</td>
<td>15</td>
<td>1.5</td>
</tr>
<tr>
<td>5</td>
<td>25</td>
<td>10</td>
<td>20</td>
<td>2.0</td>
</tr>
<tr>
<td>6</td>
<td>25</td>
<td>5</td>
<td>25</td>
<td>2.5</td>
</tr>
</tbody>
</table>

To each solution 100 ml of absolute alcohol are added. They are then stood for 1 hour and filtered under vacuum on a Buchner funnel using a 5.5 cm Whatman No. 5 paper precoated with a further 2 g of kieselguhr. The precipitate is washed with 80% alcohol, transferred to a beaker (without the filter paper), and 40 ml of calcium chloride solution added. The whole is boiled gently for 15 minutes to dissolve the starch, cooled and transferred to a 100 ml flask. After making to volume and adding a further 1.7 ml of water to correct for the volume of kieselguhr, the contents are filtered, discarding at least the first 40 ml of filtrate. 10 ml of the remaining filtrate are pipetted into a 50 ml flask and 2.5 ml of 2 N acetic acid, 0.5 ml 10% potassium iodide and 5.0 ml M/600 potassium iodate solution added. The flasks are made to volume, the contents mixed and the absorbance read at 600 nm in a 1 cm cell against the reagent blank. The graph is prepared by plotting the absorbance against milligrams of starch per 50 ml.

To test raw sugar, 25 g of the sample are weighed into a beaker and dissolved in 30 ml of warm water. 100 ml of absolute alcohol and 2 g of kieselguhr are added and the same procedure carried out as for the standard solutions up to the point of making up to 100 ml, adding 1.7 ml of extra water, filtering and discarding the first 40 ml of filtrate. 20 ml of the remaining filtrate are pipetted into a 50 ml flask, and 10 ml water, 2.5 ml 2 N acetic acid, 0.5 ml 10% potassium iodide solution and 5.0 ml M/600 potassium iodate solution added. The volume is made up, the contents mixed and the absorbance at 600 nm in a 1 cm cell is read against a reagent blank. Then starch in sugar (p.p.m.) = mg of starch from graph × 200.

(b) Tate & Lyle method\textsuperscript{14}. The reagents required are a molar solution of hydrochloric acid, methylated spirit, benzene, 2 N acetic acid, M/600 potassium iodate, 10% potassium iodide and a solution of calcium chloride, sp. gr. 1.3, prepared by dissolving 62 g of calcium chloride in 138 ml of distilled water. 100 g of the sugar are dissolved in 100 ml of distilled water and treated with 20 ml dilute hydrochloric acid and 300 ml methylated spirit. After standing for 2 hours to allow starch precipitation to take place, 2 g of filter aid are added and the suspension is filtered under vacuum in a Buchner funnel on a paper previously coated with 1 g of filter aid. The residue is washed with 100 ml of

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75% methylated spirit—25% water mixture, followed by 50 ml of hot undiluted methylated spirit and 50 ml of hot benzene. The cake is transferred to a 250 ml beaker, 50 ml of calcium chloride solution added, the contents boiled gently for 15 minutes and then allowed to cool before being transferred to a 100 ml flask, to which, after making to the mark, 1.7 ml excess water are added. The contents of the flask are mixed thoroughly and filtered and an accurate aliquot (volume dependent on the expected starch content) is transferred to a 100 ml flask, diluted to approximately 50 ml, then treated in sequence with 5 ml of acetic acid, 1 ml potassium iodide solution and 10 ml potassium iodate solution. The solution is diluted to the mark with distilled water, and after mixing the absorbance is measured at 600 nm in a 1 cm cell. The starch content of the aliquot is obtained by reference to a standard graph prepared from sucrose solutions containing known amounts of analytical grade soluble starch. The figure obtained from the graph must be divided by the volume of the aliquot taken, and multiplied by one hundred.

(c) Colonial Sugar Refining Company method. The reagents required are a 40% w/w solution of anhydrous calcium chloride; acetic acid (1 N and 0.1 N); calcium chloride–acetic acid reagent prepared by adjusting the pH of the calcium chloride solution to 3.0 with 0.1 N acetic acid; 0.01 M potassium iodate; 10% potassium iodide; a mixture of the two latter (prepared the day it is required) made by mixing 1 volume of potassium iodide with 9 volumes of distilled water and 10 volumes of potassium iodate; analytical grade potato starch and pure sucrose. From the starch, a standard starch solution is prepared containing 0.4 g of anhydrous starch per litre. This is used to prepare standard graphs according to a closely detailed procedure which can be obtained by reference to the original article. To measure the starch content of raw sugar, 40 g of the sugar are transferred to a 100 ml flask, the sugar dissolved in distilled water and the solution adjusted to volume. 15 ml of this solution are pipetted into a 50 ml flask and 25 ml of the calcium chloride–acetic acid reagent added. After mixing, the solution is maintained at 95–100°C for 15 minutes with agitation after 5 and 10 minutes heating. After cooling, the volume is adjusted to 50 ml with distilled water and the contents mixed. 2 x 15 ml aliquots are pipetted into two separate 25 ml volumetric flasks. One is to be used as a blank and the other as the test solution. 2.5 ml of the 1 N acetic acid are added to both and the blank is made to the mark. 5 ml of the iodide–iodate mixture are added to the test solution and it also is adjusted to volume and mixed. The absorbance of both solutions are read in a 1 cm cell at 700 nm and, after any adjustment for the blank, the starch content of the test solution is read from a previously prepared standard graph.

F. Affination and gristing

By affination is meant the technique used to prepare laboratory samples of raw sugar for grist measurement. A satisfactory method is that described by Bean for use by the California and Hawaiian Sugar Company. In this process,
the syrup film is removed from the sugar crystal by mixing it several times in a rotating jar with a nearly saturated sugar syrup and removing the residual film by washing with methanol and iso-propanol.

The materials required are granulated sugar syrup made to 66.0% solids at 20°C, 100% methanol and 100% iso-propanol. The apparatus consists basically of a suitable jar, with a wide mouth which can be capped, of about half a gallon capacity and a suitable roller mill for rotating the jar at a fixed speed (C & H = 275 r.p.m.). 300 g of the sugar are placed in the jar which contains 450 ml of syrup at room temperature (20°C), the jar capped and shaken by hand to mix the sugar and syrup and then mixed on the mill for 3 minutes. The mixture is then poured into a Buchner funnel containing a suitable Monel metal screen and drained under 26 in. of vacuum for 2 minutes. The sugar is returned to the jar and the operation repeated until it has been washed four times, using the same funnel and jar each time without washing. After the fourth washing all the sugar is returned to a similar clean dry jar, adding 750 ml of methanol, and the whole is mixed for three minutes. After filtration the washing is repeated with 750 ml of iso-propanol. Filter again and transfer the sugar to a flask or jar and dry on a water-bath until no odour of alcohol can be detected. The sugar is now ready for grist analysis and this can be done in two ways; either by separation on standard sieves (B.S.S. or Tyler), expressing the amount through one particular mesh size as a percentage of the whole, or by the method of Powers 16, which defines the sugar in terms of mean aperture (M.A.) and coefficient of variation (C.V.). The mean aperture is the size of mesh through which 50% by weight of the sample will pass, and the coefficient of variation expresses the deviation from this mean size according to the formula:

\[
C.V. = \frac{\text{Standard deviation} \times 100}{\text{M.A.}}
\]

Both methods can be used satisfactorily.

G. Colour

The American No. 10 contract lays down a colour specification in terms of ICUMSA method 2 of 195817, while the suggested British Standards Specification judges colour by method 1A, also of 1958. Details of the two methods can be found by reference to the original source. However, it should be noted that, at the time, ICUMSA was indulging in the luxury of having no less than seven tentative methods, all based on the measurement of absorbance by various methods. These methods have gradually been whittled down until finally, at the most recent (1974) session, an attempt was made to reduce the number to one official method, based on the measurement of absorbance at a single wavelength18. The attempt was unsuccessful and method 2 of 1958 remains in being at least temporarily. There seems little doubt, however, that eventually agreement will be reached on a uniform method for determining colour.

ACKNOWLEDGEMENTS

In preparing this review, the authors were helped by the constructive advice of their colleagues, Dr. K. J. Parker and Mr. J. M. Hutson, and would like to acknowledge their valuable contribution.

SUMMARY

There is a relationship between the type of raw cane sugar taken into a refinery and the difficulties encountered in turning it into good quality white sugar. Since the turn of the century, an increasing amount of attention has been paid to the situation, as reflected by the volume of publication. This literature, comprising individual papers and the extensive deliberations of the numerous national and international organisations interested in sugar, has been reviewed.

The effect of variations in raw sugar quality on the separate unit operations in refining is discussed in detail, with emphasis on storage, affination, defecation, filtration, decolorisation and recovery. Resulting from these, the need and reasons for setting standards for raw sugar quality are set out along with indications of the characteristic properties to be monitored.

Since a buyer-seller relation usually exists between the raw sugar producer and the sugar refiner, a satisfactory method of evaluating the economic cost of variations in the critical properties of raw sugar has had to be worked out. The fixing of datum levels from which penalties and premiums are calculated is important. Experience has played a major role in this context, but, whatever is agreed, it must be worthwhile for the producer not to transgress the limits, if only to make it unnecessary for the refiner to invoke the penalty clauses. Those factors most likely to form the basis of a contract are discussed, with particular reference to polarisation, moisture, inorganic impurity and ease of refining. The calculation of realistic cost-quality relations is considered in some detail with examples of practical scales. Polarisation is usually based on a datum of 96° pol with a moisture around 1%. The ash impurity is an important melassigenic factor which determines the acceptable ash level in the region of 0.6%. Although filterability is important to the refiner, it is difficult to measure its effect satisfactorily, as witnessed by the plurality of methods cited.

The manner in which different producers have tackled the production of raw sugar of satisfactory quality is expounded. The control of polysaccharides has featured extensively. Probably the most important factor under the control of the producer influencing the refinability is the crystal quality—shape, size and size distribution. It is concluded that there has been a marked improvement in these standards, despite the introduction of continuous centrifuges whose method of operation could quite easily have been disastrous to subsequent refining.

Finally, the methods of measuring the quality of raw cane sugar are reviewed. In the main they are a reflection of the achievements of ICUMSA in Sugar Technol. Rev., 3 (1975) 69–126.
analitical standardisation. The most difficult area is clearly filterability, which is discussed in depth.

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Hay relación entre el tipo de azúcar crudo que recibe la Refinería y las dificultades que se encuentran para convertirlo en azúcar blanco de buena calidad. Desde principios de siglo se ha dado una atención cada vez mayor a este aspecto como se refleja en numerosas publicaciones. Esta literatura incluye tanto trabajos individuales, cuanto extensas deliberaciones de numerosas organizaciones interesadas en el azúcar, tanto nacionales como internacionales que han sido revisadas.

Se discute en detalle el efecto de las variaciones en la calidad del azúcar crudo en las distintas operaciones de la refinación, con énfasis en almacenamiento, afinación, defecación, filtración, decoloración y recuperación. Como resultado de todo se deriva la necesidad de establecer estándares para la calidad de azúcar crudo a la vez que las indicaciones de las propiedades características que se deben juzgar.

Puesto que generalmente una relación comprador-vendedor entre el productor de azúcar crudo y el refinador, se deberá idear un método satisfactorio para evaluar el costo económico de las variaciones en las propiedades del azúcar. Los niveles de base para los castigos y premios se calculan y son importantes. La experiencia ha jugado un papel mayor a este respecto, pero, cualquier cosa que se acuerde debe valer la pena para el productor y que no exceda los límites y haga innecesario para el refinador invocar las cláusulas de castigo. Estos factores deben formar la base de un contrato para ser discutido con referencia particular a pularización, humedad, impurezas inorgánicas y facilidad de refinación. El cálculo de las relaciones costo real/calidad se considera con algún detalle con ejemplos de escalas prácticas. La fabricación se basa generalmente en un nivel de 96° Pol., con una humedad alrededor de 1%. Las impurezas son un factor melásigeno importante
que determina la aceptación de un nivel de cenizas en una región del 0.6%. Aún cuando la filtrabilidad es importante para el refinador, es difícil medir satisfactoriamente su efectividad como testigo por la pluralidad de los métodos citados.

Se cita con amplitud la manera como los diferentes productores han calificado la producción de azúcar crudo de calidad satisfactoria. El control de los polisacáridos se ha detallado extensamente. Probablemente el factor más importante bajo el control del productor que influye en la refinabilidad es la calidad y forma del cristal, su tamaño y la distribución del tamaño. Se concluye que se ha obtenido un mejoramiento marcado en estos estándares a pesar de la introducción del centrífugado continuo, cuyos métodos de operación pudieran haber sido fácilmente desastrosos a las refinaciones subsecuentes.

Finalmente, se revisan los métodos para medir la calidad del azúcar crudo que principalmente son un reflejo de los logros del JCUMSA* en la estandarización analítica. El área más difícil es, indudablemente, la filtrabilidad que se discute a fondo.