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IN-LINE MEASUREMENT OF COLOR IN RAW AND REFINED SUGARS.

AUTHORS

Margaret A. Clarke[1], Leslie A. Edye[1], and Bjarne Chr. Nielsen[2] .

[1]Sugar Processing Research Institute, Inc., 1100 Robert E. Lee Blvd., New Orleans, LA, USA 70124, [2]Neltec Denmark A/S, Egegaardsvej 2, DK-6541 Bevtoft, Denmark

ABSTRACT

An instrument is described that can measure colour rapidly on solid, crystalline sugar, wet or dry, and with good correlation to the ICUMSA colour measurement at 420nm. Sugar colour can be measured in a few seconds, on sugar moving on a belt, or in a screw or a hopper conveyor. The system has been used on white crystal sugar, and offers rapid control of sugar going to packaging or storage. The system can be applied to monitor colour on sugar coming out of batch centrifuges, for a new level of quality control. Recent work has shown that the system can be used to measure colour in raw sugars, and in soft brown or yellow sugars. Results are reported and discussed.

INTRODUCTION

Measurement of sugar colour is important for product quality control and for process control in refineries and factories that produce white sugar. In North America, the colour measurement in solution form (either ICUMSA colour (4) or Reference Base Units) provides common ground between sugar manufacturers and their customers; there is no measurement of colour in solid crystalline white sugar that is widely used for white or raw sugars. Reflectance measurements are available (4) (Braunschweig colour standards) and are in use in Europe, but are not in wide use in North America, because they correlate poorly to the established colour measurements.

Colour measurement is important in process control as a measure of process efficiency and of product quality. Colour content correlates roughly with ash, moisture and non-sugars content; there are rapid tests for ash and moisture, but not for colour. Colour of final product is almost always included in customer specifications. Indeed, in recent years a specification based on ICUMSA colour has been added to white sugar contracts traded on the futures market.

Sugar of colour above specifications must be recycled in the refinery or factory at considerable cost, or may be returned by the customer at even more expense. In production of crystalsugar, good controls are available for crystallisation, and size and yield of crystals. However, for the subsequent separation of sugar crystals by centrifugation, and washing and drying, few controls or tests are available without expensive use of labour. And so the final preparation of white sugar, at its most cost-sensitive stage, is run without frequent monitoring or inspection.

A rapid, reproducible on-line measurement for colour in solid sugar, that correlates to the accepted solution colour, is desirable. A system to make such a measurement has recently become available. The multiwavelength instrument from Neltec Denmark can measure colour of sugar in less than 5 seconds, on a moving belt or conveyor, with no sample preparation. (1,2). The instrument can be used to measure colour, in terms of up to 5 different calibrations, in wet or dry sugar, in a screw or hopper conveyor or on a belt.

The system has been installed on white sugar lines to check colour of wet white sugar coming from centrifugals (1, 3), so that the performance of each centrifugal can be monitored automatically.

Advantages of this system are:

1. The risks of bad sugar in the drier or the silo are eliminated, as is the risk of inadvertently shipping sugar with too high a colour value.
2. Better control of centrifugals. Malfunctioning is reported immediately. As it is possible to trace which centrifugal is not working properly, response can follow without delay. Stops in the production due to centrifugal problems can be avoided or the consequences can be greatly reduced.
3. Trimming of washing times. The fast feed-back on changes in washing times for each centrifugal makes trimming easy. With continuous monitoring of quality, the security distance to the quality limit can be reduced, leading to energy savings.
4. Speed. The results are presented less than 5 seconds.
5. No sample preparation is required. The measurements are taken directly in the production stream. The frequency of the laboratory tests can be reduced, with labour savings.
6. The quality is improved. More uniform quality is better quality.

In the present paper, successful applications of the Neltec system of colour measurement of raw cane sugars and of soft brown or yellow sugars are reported. Applications to white sugar colour measurement are reviewed. In the case of soft sugars, calibrations can be made against ICUMSA colour or against colour from the AGTRON ™ measurement that is popular in North America, to give a continuous analysis of soft sugar colour, one of the major customer specifications for soft sugars. The system can be applied to boiled soft sugars or to those prepared by a coating and blending process.

Raw sugar colour can be measured continuously on raw sugar entering a refinery, or, and perhaps more significantly, on washed raw sugar coming out of the affination centrifugals. Until now, there has not been (to the authors' knowledge) a system for continuous measurement of washed raw sugar colour, which is probably the most important parameter for process control in a refinery. The colour of washed raw sugar, coming out of the centrifugals, can be monitored every few seconds by the Neltec system.

METHODS AND MATERIALS

Measurement System

The basic measurement unit is working on a very simple principle (Figure 1). An illuminator sends out short pulses of white light. A detector collects some of the reflected light and separates it in spectral components. Both units have housings of stainless steel, and are placed at a distance of about 1 meter from the sugar. Therefore they can resist the environmental conditions, and are not contaminated by the sugar.

Information about the reflected light is sent from the detector to a computer, where the colour is calculated and presented to the user. Two graphs are used, a short term graph showing colour during the last 30 minutes (Figure 2), and a long term graph to show the last 24 hours (Figure 3). Under the short term graph is shown information about which centrifugal(s) has delivered sugar to the measurement position at the time of measurement (Figure 4). In this way the operator is able to identify centrifugals with unsatisfactory function.

Calibration of the ColourQ 800

The system must go through two calibrations, one initial and one check calibration at regular intervals. The initial calibration is made after the installation. A number of samples are taken from the normal production stream. Their colours are determined in the laboratory. These results are combined with the measurements from the system to give a calibration curve. This is then implemented in the system. Later during normal operation a check calibration is needed to compensate for drift in the instrument. This is made simply by measuring a white tile at regular intervals.

Laboratory Installation and Operation

Although the Neltec ColourQ 800 is designed to operate in a factory environment with sugar moving on conveyor belts or screws, the laboratory set up for calibration development, validation and precision tests used stationary sugar samples. The Neltec light source and detector were mounted on a scaffold (Figure 5) above a sample tray (25cm wide x 75cm long x 7cm deep with a white enamel inside surface facing the detector). The white sugar samples were loaded onto the tray with a device that simulates sugar falling onto a conveyor belt. For raw sugars and soft sugars a brown paper liner was placed inside the sample tray and the samples were loaded by hand. The surface of the sample was between 70 and 85 cm below the light source and detector.

Calibration and Validation at SPRI Laboratories

Calibrations for raw sugar were developed from Neltec measurements and conventional colour measurements (ICUMSA, both buffered and old unbuffered methods) obtained at SPRI. Calibrations for refined cane sugar were developed from Neltec measurements obtained at SPRI and conventional colour measurements (ICUMSA, buffered method) obtained from the refinery supplying the sugar. Calibrations for soft sugar were developed from Neltec measurements obtained at SPRI and conventional colour measurements (ICUMSA, buffered and old unbuffered methods) obtained at SPRI and AGTRON™ colour measurements obtained from the refinery supplying the sugar.

Multiple linear regression or neural network methods were used to develop the calibration models. The validation data was obtained at SPRI.

RESULTS

Details on the data sets used to develop the models are shown in Table 1.

A. White sugars - dry

In a production environment the sugar is measured on a moving conveyor belt, where it is measured as a continuous flow with an even surface. In the laboratory measurements the surface is less even, because the sugar is loaded on the sample tray by a hand-operated device. To test the influence of this the validation was repeated.

The results are shown in Table 2 and Figure 6. The figure has data from the first validation only. The average colour measured by the refinery was 39.0, versus 37.5 and 39.3 by the two validations.

It can be observed that the precision of the laboratory measurements (SEP 3.1 and 2.8 by the two validations) is not as narrow (SEP 2.23) as reported (3) for measurements in a production environment. Apparently the smaller sample size and the hand loading have a negative effect on the precision of the measurement. The precision of the production on-line measurement compares very well with the repeatability of the ICUMSA solution colour measurement of 3 ICU (repeatability: same operator, same sample) (4).

B. Raw sugars

The calibration was made on the SPRI Raw Sugar Library on sugars with ICU up to 15,457.

The results are shown in Figure 7. The average of the ICU is 4635 and 4992 of the prediction. SEP is 933. The SEP appears to be high; in fact, for 13 of the 89 samples the Neltec and ICUMSA colour differed by more than 1,000 ICU (residuals > 1,000 ICU) and, of course, these samples inflate the SEP value. It is important to note that in this and other studies a visual ranking of raw sugar colour intensity often does not agree completely with a ranking by the ICUMSA measurement. This difference is related to the proportions of various classes of colorants in raw sugars (7, 8) and to the sensitivity of the human eye to light at 420nm; a more detailed explanation is beyond the scope of this report. Comparison of ICUMSA and visual ranking of the raw sugar library indicated all samples that ranked differently had Neltec-ICUMSA residuals > 1,000 ICU. That is, that in every case when the Neltec measurement had > 1,000 ICU difference from the ICUMSA colour value, the visual colour ranking disagreed with the ICUMSA ranking. Furthermore, the ranking of raw sugar colour by the Neltec agreed with the visual ranking rather than with the ICUMSA ranking.

The SPRI library of raw sugars covers the widest possible range of quality, colour, and grain size, from very light VHP (Very High Purity; highly washed) sugars with minimum syrup coating, to fine grain sugars with a lot of molasses coating that have been stored for years. The error in ICUMSA colour measurement on this set is much greater than the error of 200 to 400 on a set of narrower range (eq. 6000 to 8000 ICU). Sources of the ICUMSA error in measurement of raw sugar colour are many in addition to difference in crystal coating. The filtration step filters out a non-uniform amount of colour from each sugar (5, 6). The higher molecular weight colour is more likely to be filtered out on a Millipore-type membrane. However, this is the very colour that continues to persist into the refined sugar crystal, because no refinery filtration will simulate the Millipore effect.

Errors in the raw sugar colour measurement by ColourQ have to have the same range for accuracy as the ICU measurements, since the Neltec System is calibrated against ICU, but the ColourQ shows much better precision than the ICUMSA measurement, as shown in Table 3. Repeatability (precision) is an important factor in the continuous measurement of raw sugar colour.

Because the Neltec system can be used on wet or dry sugars, it is proposed that the system be used to measure colour of washed raw sugar coming out of the affination centrifugals. Trials on this proposed system are now taking place. Colour of washed raw sugar is a most important control factor for any refinery; knowledge of WRS colour allows predictions for control of clarification and decolorisation. To the authors' knowledge, there is no continuous monitor for washed raw sugar colour currently in use. Most refineries take this measurement once every 4 or even 8 hours - and yet it is a most significant basis for refinery control. Problems in measuring colour over a wide range of colour in whole raw sugars, as in the SPRI library of raw sugars, are far diminished, because:

1. Range of colour on washed raw is relatively narrow, compared to the library. Range will vary in depth according to refinery policy, that is, whether a single raw is melted at a time or whether raws are blended. Overwashing or underwashing of raw will, however, stand out when colours from individual centrifugal dumps are continuously monitored.
2. Depth of syrup coating on crystal will be reduced on washed raws and will contribute much less to error than in calibrations for whole raw sugars.

C. Soft sugars

The calibration was made on soft brown sugars with AGTRON™ values in the range of 19-61, and ICU in the range of 1176-10049.

The validation for the calibration corresponding to AGTRON™ values is shown in Figure 8. The average of the AGTRON™ values is 45.6, and 43.7 of the prediction. SEP is 2.4. The validation was made almost 2 months after the calibration data were obtained. The validation show lower values from the ColourQ than the original AGTRON™ values, indicating that colours were darker at time of validation than at time of calibration. This darkening of soft sugars with time, is a well known phenomena in both light and dark soft sugars, with the colour increase generally correlated directly to moisture content of the soft sugar (9).

The validation for the ICU values is shown in Figure 9. The average of the ICU is 4292, and 3961 of the prediction. SEP is 929. To test the influence of the samples with high ICU values, all samples with ICU greater than 7000 were excluded from the validation set. This reduced validation set was measured with the same calibration. On this reduced set the average of the ICU is 3291, and 3121 of the prediction. SEP is 527.

One measurement system can have both AGTRON™ and ICUMSA calibrations installed, and can measure both values for simultaneous presentation.

From the comparison of laboratory and production line measurements on white sugars, the following prediction may be made: Because measurements on soft brown sugars were made with the ColourQ 800 only in a laboratory system, and not on an on-line production system, it is to be expected that the precision of the results will be wider (not as good precision) than would be found in an on-line measurement - that is, the laboratory measurement is “worst case” - an unusual situation, but understandable because of the difference in sample size.

Precision tests

The repeatability of the ColourQ 800 was tested by measuring a sample 10 times for both raw and soft brown sugars. For comparison a sample of raw sugar was tested 9 times by the conventional ICUMSA laboratory procedure. The results are shown in Table 3. The ICUMSA guideline (4) for repeatability of raw sugar is <110 for sugars 500 to 2000 ICU and <300 for sugars 2000 to 7000 ICU.

CONCLUSIONS

The automated, on-line Neltec system can give rapid, reliable and reproducible measurements for colour of white, raw, and brown sugars. Comparison of the system with conventional laboratory measurements is summarised in Table 4, for time, accuracy, precision and degree of manpower.

1. For white sugars, the current study has confirmed that the Neltec system can be used to give accurate and precise colour measurement. It can provide an almost continuous analysis of wet sugar coming from centrifugals, thereby establishing a constant monitor for centrifugal performance and reduce costly recycling. It can monitor dry white sugar, out of driers or silo, thereby reducing out-of-specification problems and customer complaints and returns.
2. On raw sugars, this study has shown that the Neltec system can give colour measurement comparable to the ICUMSA measurement, under the worst conditions (widest range of raw sugar colour and quality). The speed of the Neltec system measurement allows an almost continuous analysis of raw sugar entering a refinery (or exiting a factory). The ability of the Neltec system to measure colour of wet sugar means that it can be used to monitor washed raw sugar colour in a refinery, monitoring centrifuge performance, and thereby reducing overwashing in affination with consequent energy savings.

It can equally well be applied to measure raw sugar colour in a raw sugar factory (or blending station where VHP raws are coated), offering continuous control of raw sugar colour, with similar benefits for centrifugal control. Problems in centrifugal operation can be identified in a few seconds. Wash levels can be minimised, and amounts of sugar lost to A or B molasses can be reduced. For raw sugar factories producing a high grade edible speciality product, the indication that the Neltec instrument correlates better with visible colour than does the ICUMSA measurement may be a useful property.

1. For soft sugars, the Neltec instrument offers the opportunity for rapid measurement of each batch of sugar boiled, or a continuous measurement of colour in blended (coated) sugars. The colour can be read simultaneously in both ICUMSA units and the AGTRON™ units popular in North America. There is space for 5 calibrations (or simultaneous readings) so a calibration for each of the various types of AGTRON ™ instruments could, if wished, be used to satisfy requirements of different customers. In summary, the Neltec system can be used on-line in the raw sugar side and the final product side (white or brown) of a refinery to provide rapid, reliable measurement of colour.

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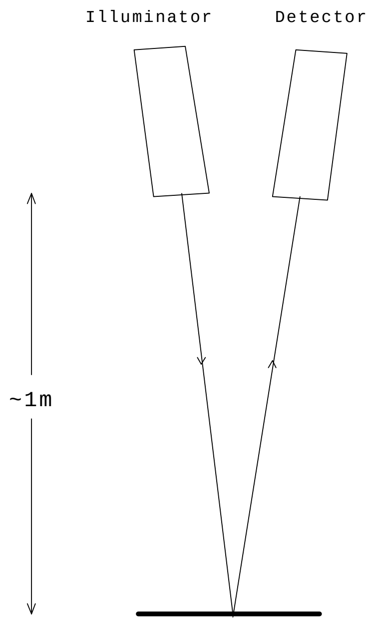

Figure 1. Position of illuminator and detector over sugar.

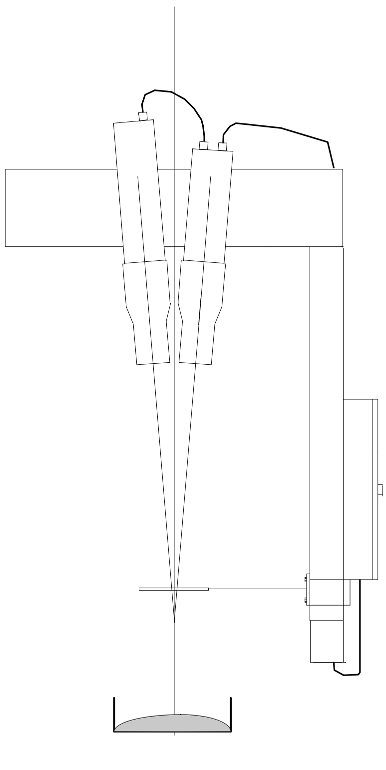
Figure 5. Measurement of dry sugar in sample tray

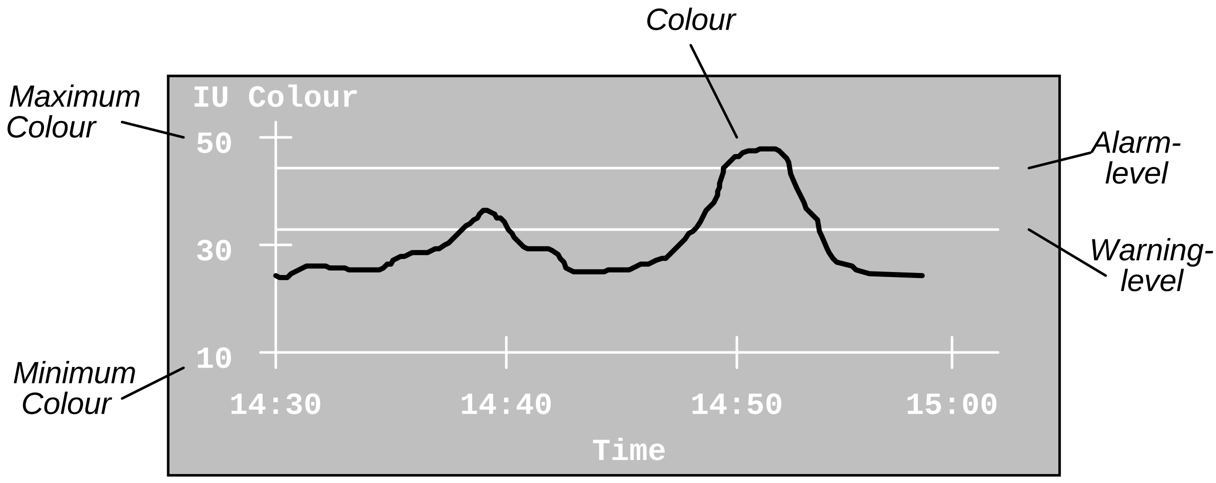

Figure 2. Short term graphics

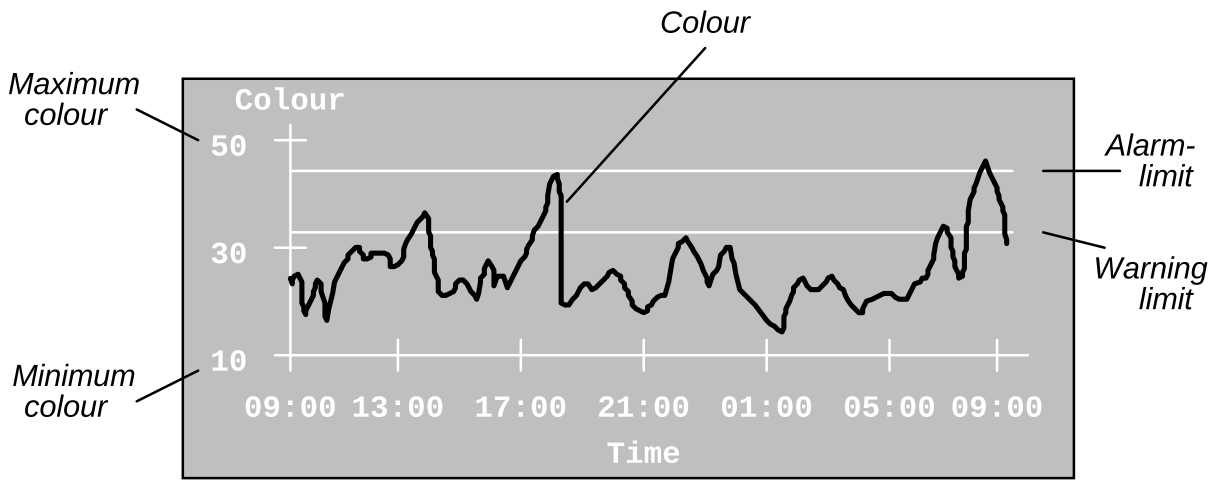

Figure 3. Long term graphics

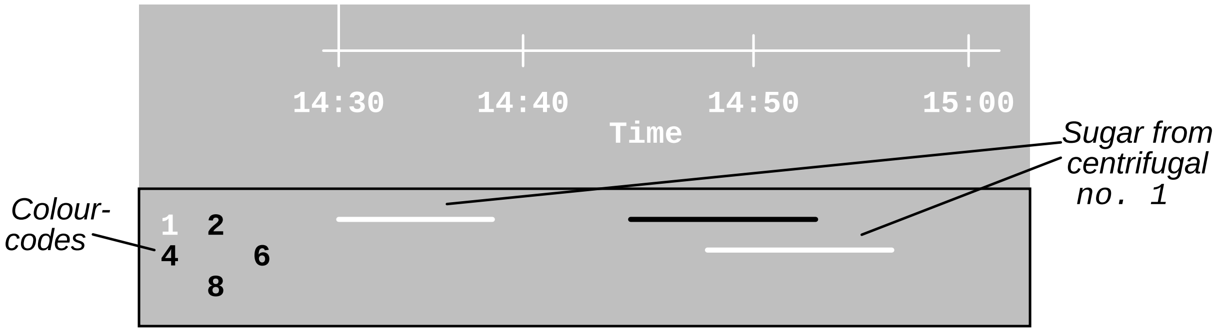

Figure 4. Identification of centrifugals

	CALIBRATION			VALIDATION		
	# samples	Mean	Range	# samples	Mean	Range
Raw sugar (ICUMSA, buffered)	89	4744	238 - 15457	14	4634	2299 - 6145
Refined cane sugar (ICUMSA, buffered)	50	41.1	7.9 - 73.6	16	39	7.4 - 64.0
Soft Sugar (AGTROM TM)	55	44.8	19 - 61	18	45.5	19 - 61
Beet white sugar (ICUMSA, buffered)	234	34	25.0 - 104.8	N/A.	N/A.	N/A.

Table 1

Samples for calibration and validation

Buffered		
Refinery lab	Neltec	Neltec Rep.
7.4	7.9	7.7
15.5	12.0	13.5
29.1	26.3	29.9
24.5	21.8	25.0
34.3	29.0	33.1
34.4	35.1	38.1
45.8	43.4	48.0
45.9	44.9	49.5
53.0	51.3	53.2
56.6	53.7	53.0
48.8	49.5	49.0
57.2	50.3	50.8
19.5	22.6	23.9
30.8	34.5	33.9
57.1	55.3	55.0
64.0	62.7	64.8
SEP	3.1	2.8

Table 2

White cane sugar from refinery

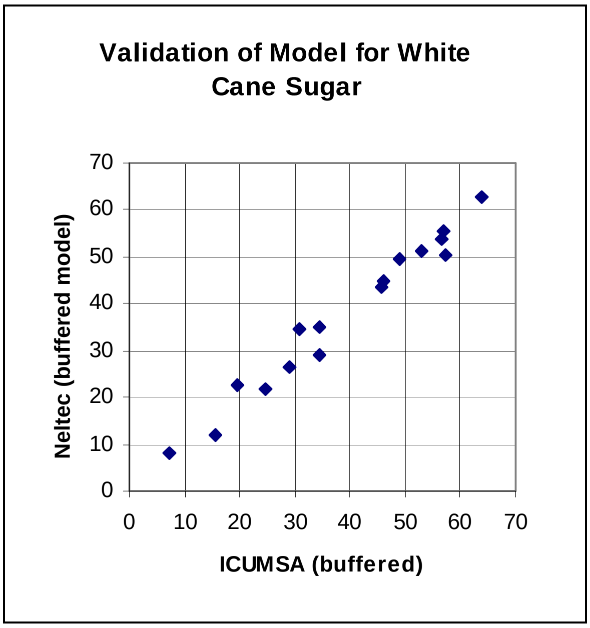

Figure 6

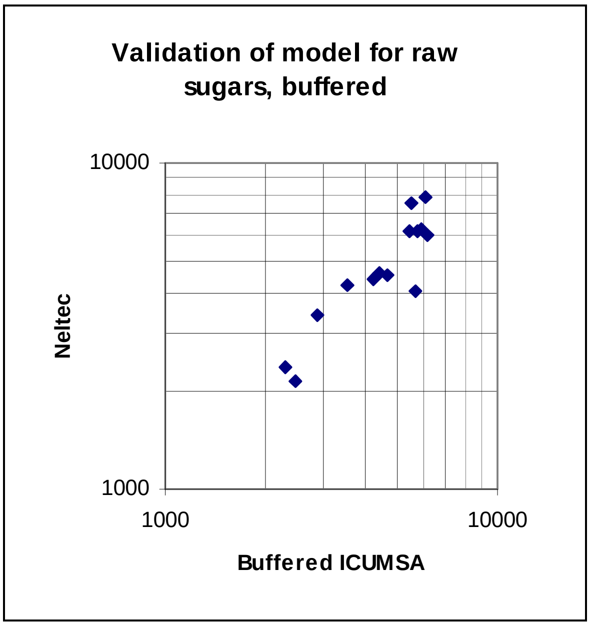

Figure 7

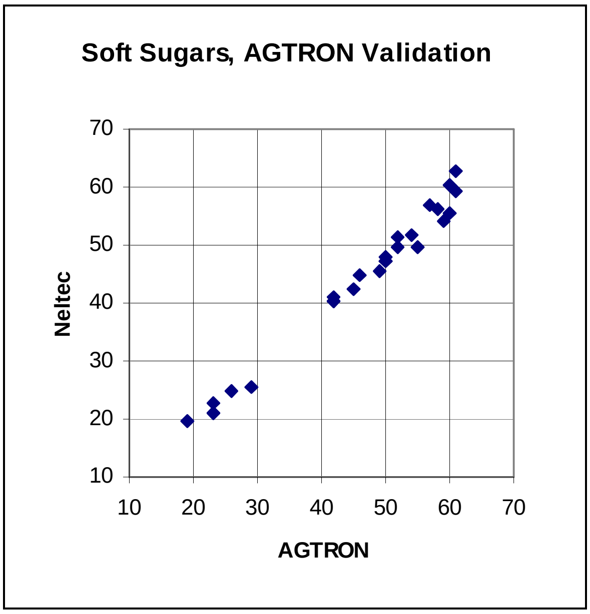

Figure 8

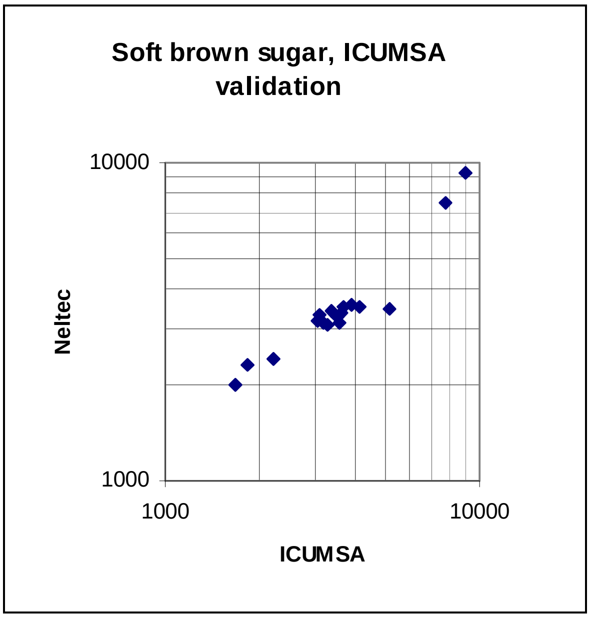

Figure 9

Repeats	RAWS				SOFTS	
	ICUMSA by Neltec	abs residual	ICUMSA by Lab	abs residual	AGTRON™ by Neltec	abs residual
1	3239	31	3073	155	24.4	0.93
2	3205	65	3013	95	22.4	1.07
3	3305	35	2941	23	23.4	0.07
4	3289	19	3038	120	22.8	0.67
5	3305	35	2962	44	23.8	0.33
6	3317	47	2873	45	23.7	0.23
7	3233	37	2775	143	22.9	0.57
8	3250	20	2785	133	24.0	0.53
9	3283	13	2805	113	23.6	0.13
10	3274	4			23.7	0.23
mean	3270	31	2918	97	23.5	0.28
Std. dev.	37	18	113	48	0.6	0.34

Table 3

Precision test - statistical analysis of 10 replicates

		Standard (laboratory)	Neltec
Time	ICUMSA colour	15-20 min	5sec
	RBU	6-10 min	5sec
	AGTRON™	2-4 min	5sec
Accuracy	White	±2 ICU	Similar but larger than method used for calibration
	Raw	±100 ICU	
Precision	White	3	2.9
	Raw	113 (100-300)	37
	Soft	1.0	0.6
Labour level		Intensive, requires training	Very low

Table 4.

Comparison of standard colour measurement
with Neltec system measurements.