CONTROLLING QUALITY OF MDS EXTRACT FOR LONG TERM STORAGE

David R. Groom, Terry D. McGillivray and James H. Heggeness
American Crystal Sugar Company, Technical Services Center,
PO Box 1227, Moorhead, MN 56561-1227

Introduction:
American Crystal Sugar Company (ACSC) has two molasses desugarization (MDS) facilities. One facility is at East Grand Forks (EGF), Minnesota, commissioned in the fall of 1993. The EGF facility is a "standard" simulated moving bed (SMB) separator that produces two fractions, a sugar rich extract, and a by-product raffinate. In January of 2000 ACSC commissioned a second MDS facility at Hillsboro (HLB), North Dakota. The separator utilizes displacement chromatography along with simulated moving bed technology. It has been the practice of ACSC to store the extract juice from each desugarization facility for up to 12 months and then process the stored extract juice after completion of the beet processing campaign. In recent years, we also co-process (blend extract with beet thick juice) during part of the beet campaign at several factory sites. This paper will review past challenges and the resolution of problems. Specifically, this paper will focus on the production and storage of extract at Hillsboro's MDS facility.

In the 2003 and 2005 ASSBT conferences, the authors presented papers that covered the resolution of long term storage issues encountered at Hillsboro. Changes made to the operation of the MDS separator produced extract that was stable in storage. In the last 4 campaigns, color rise in storage has been minimal, pH has been stable, and purity has remained unchanged for as long as 14 months. Hillsboro extract has typically been sent to storage with a minimum RDS of 68.5, a temperature below 20°C, and a pH of 10.0. With a history of successful long term storage, the pH target has been dropped to 0.5 units to 9.5. Overall color removal on a mass basis has been 85 to 88 percent, resulting in extract color that was 16 to 20 percent of feed molasses color.

Since extract has been storing well, the focus on increasing recoverable sugar from extract has shifted to reducing extract color. At both EGF and HLB factories, throughput from extract is 50 to 70 % that during beet slice campaign. Over the last 4 years, daily white sugar production rates have improved from 9,000 to 20,000 hundredweight (cwt) per day. The 20,000 cwt. per day production rate is substantially less than the 32,000 cwt typically maintained during beet slice campaign. The predominant factor causing reduced throughput is elevated color of the extract coming into the sugar end. Consequently, the standard liquor feed to the white pans will have a high color and high purity. The color to nonsugar ratio of HLB standard liquor has been above 1500 during extract campaign. Even after reducing extract color with carbonation, the color to nonsugar ratio in standard liquor is at best 900. In contrast, standard liquor produced from typical beet thick juice has a color to nonsugar ratio of 450 to 550. The elevated ratio of color to nonsugars makes it difficult to produce white sugar with 3 stage boiling.

Data were retrieved from a recent HLB processing campaign (2004-2005). Principal components analysis was used to build a sugar end production model. One of the major components used to predict throughput was standard liquor color. Based on the model, a 4000

ICUMSA color rise in standard liquor would result in a 7000 cwt drop in sugar end capacity. Given the major impact of color on processing throughput, the focus shifted to the source(s) of color well before the MDS plant.

Currently, there are two areas of focus to reduce color. One area of continuing focus is the molasses that feeds MDS. In turn, the molasses itself is influenced by the quality of beet along with processing issues in the beet and sugar ends of the factory. Minimizing the formation of invert sugar at various processing stages is good factory practice. Not only can color be reduced in extract, but less sucrose is lost during beet slice campaign. One way ASC minimizes invert production is in the tracking of invert formation across the diffuser and keeping it to a minimum. Control of invert formation across the evaporators and in the sugar end is an ongoing effort. Minimizing invert formation in the beet and sugar ends reduces the molasses color. At ACS, factories with the lowest molasses color also have the lowest level of invert production during processing. The second area of focus is operating the MDS separator to specifically reduce extract color. Tuning of the separator can be done to reduce color in the extract; however sugar loss increases through the separator when color removal is high. The down side is that overall sugar leaving the MDS plant as extract is reduced. ACS is currently working on optimizing the process by reducing molasses color and increasing color rejection in the separators.

To quickly assess the impact that changes made on extract color, samples of extract were evaluated in accelerated storage trials (AST). Samples were kept at a temperature of 50°C during accelerated storage. One particular area of focus was the impact of invert sugar on color rise observed during storage. A subset of extract samples placed in AST were pulled and stored at ambient (22°C) temperature. Color rise and invert sugar concentration were also tracked in the ambient samples. A model was constructed relating ambient color rise to the color rise measured in AST samples.

The most rapid color rise occurred soon after the extract was placed into storage for both AST and ambient samples. The color rise inversely corresponded to a drop in measurable invert. As noted by numerous authors for technical sucrose juices, pH played a central role along with invert in the formation of color in stored extract. Trials were conducted with extract that was stored at pH 10 and contained from 0.05 to 0.1% invert sugar on dissolved solids. Under such conditions, 1 ppm invert typically corresponded to 1.2 - 1.6 units ICUMSA color rise. Initially, one day of AST was equivalent to 14 days storage at ambient temperatures. The rate of color formation decreased as invert levels dropped in the extract. The bench top color rise tests were very conservative; we observed less color rise in the factory tanks.

Summary: Invert sugar concentration plays a critical role in producing color-stable extract for long term storage. Limiting formation of invert sugar not only benefits sugar production from extract, but reduces overall processing losses during beet slice campaign. Color contribution from invert in stored extract happens relatively quickly. The rate of color formation increases rapidly with increasing temperature and pH. During the latest extract storage trials, most of the invert was destroyed within 90 to 120 days of production with a consequent increase in color. Minimizing beet end infections, proper carbonation station operation, minimizing invert formation in the sugar end, good pH control, and reducing sugar end recycle are among the tasks that ultimately reduce extract color. Tuning the MDS separators to reduce color and invert level

are additional measures that can be taken to reduce color. However, separator tuning should be considered as a secondary component in obtaining the best overall sugar recovery from molasses.

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KAWLEWSKI, RON, STEVE AAKRE, and JIM SCHUELLER, Southern Minnesota Beet Sugar Cooperative, 83550 County Road 21, Renville, MN 56284. **Methods for automated particle size analysis.**

ABSTRACT

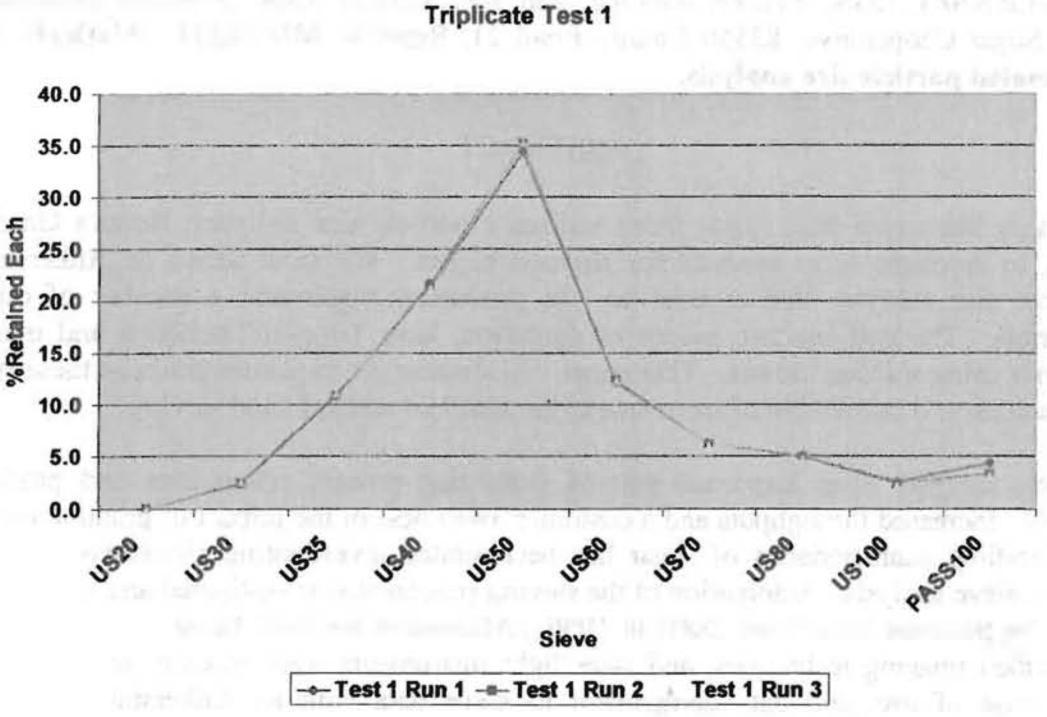
Southern Minnesota Beet Sugar Coop utilizes a particle size analyzer, Rotex's Gradex 2000, to automate sieve analysis for shipped sugars. We have added an Ankersmid particle size analyzer that is used on our production sugar and a number of other materials. The unit has two modes of operation, laser Time-of-Transition and image analysis using a video camera. This paper will discuss the implementation of these two instruments and correlation of sieve data to the standard method hand sieving.

Particle analysis is an important part of evaluating process efficiencies and product quality. Increased throughputs and a customer awareness of the impact of granulation on the handling characteristics of sugar has necessitated investigating alternatives to the classic sieve analysis. Automation of the sieving process was investigated and concluded with the purchase of a Rotex 2000 in 1996. Alternative methods based on microscopy and video imaging techniques, and laser light instruments were recently reviewed for cost, ease of use, and data comparison to sieve data with an Ankersmid CIS-100 purchased in December 2005.

Sugar is typically measured for size and distribution using a stack of woven wire test sieves. These can be used for hand sieving or placed in a mechanical shaker which incorporates a motion that moves the sample across the screens with a secondary motion. This secondary motion is usually provided by a large piece of steel on top of the stack. This tapping action will help lift the sample off the screens. The weight retained on each sieve is obtained and is typically used to calculate the Mean Aperture (MA) and Coefficient of Variation (CV). Various methods have been developed to determine these values. The Butler method is what we use which calculates the weighted average value directly from a nine sieve set without graphing of values.

Sieve analysis is the standard method referenced by ICUMSA GS2-37 and is probably the most widely used technique. The wide spread use of sieving for particle distribution is due to its low cost, reliability, and simplicity[1]. Sieving covers a wide range of particle sizes from 100 millimeters to 20 micrometers [2] and is well suited for free flowing dry powders. The reproducibility of this method can be influenced by factors such as the size of the sample charge selected, which can blind the screens. Additional factors include Percent moisture, particle shape, or agglomerates. This last factor can allow easier fracturing during subsequent bulk handling, such as pneumatic unloading, yielding a finer product.

Sugar is essentially a non-friable material under the conditions specified for sieving and will give consistent results across several test runs. Several samples were run in triplicate to test the durability of sugar under the standard gyratory motion with tapping for ten minutes.



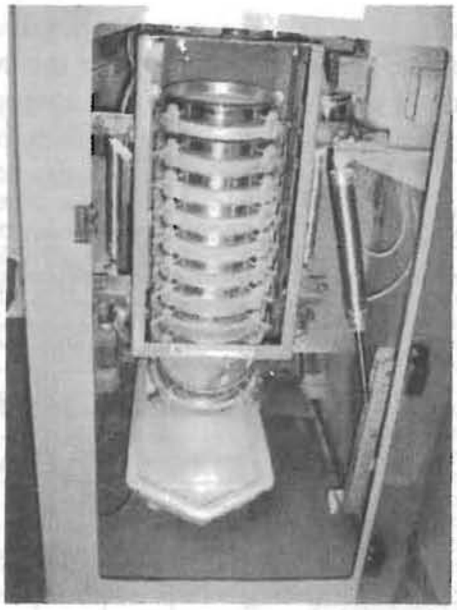
One of the principle issues with sieving by hand is retention of the sample during preparation and weighing. A low relative humidity can impact the static charge present and cause two issues. The first is the sugar particles will tend to cling to the sieves and give results biased to the coarser sieves. Second a build up of static electricity can impart the sugar particles with a tendency to repulse from each other, shooting particles out of the funnel and weighing cup. The use of metallic spoons and cups versus plastics greatly assist in diminishing this effect.

Recent years have seen increased demands for particle size analysis data. Customer requirements for certificates of analysis include ever more stringent sugar particle size requirements. The use of a mechanical shaker with manual sieve cleaning and weighing of the fractions had long been utilized. As more screen tests were required, a concern with repetitive stress injuries was becoming an issue. To resolve these issues, some way to automate sieve analysis was considered.

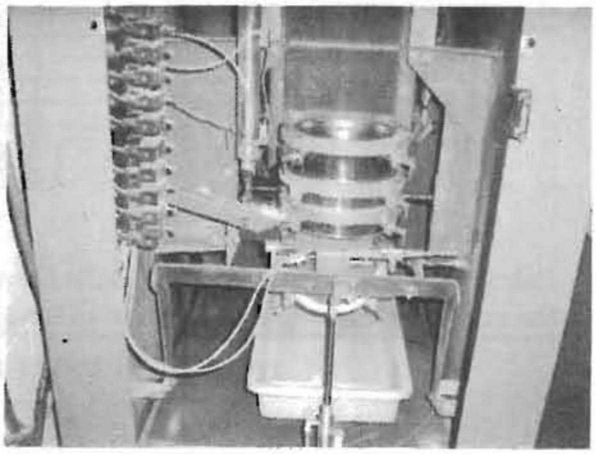
The Gradex 2000 used at Southern Minnesota Sugar is fitted with nine standard half height sieves.

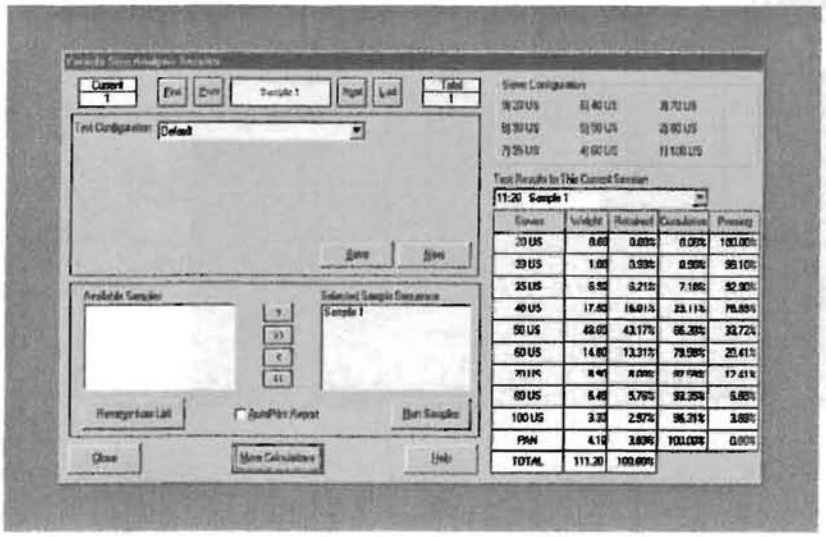
USS 20	USS 30	USS 35	USS 40	USS 50	USS 60
USS 70	USS 80	USS 100	Pass US 100		

The sieves are fitted into aluminum rings which are in turn bolted to a belt. This belt provides for attachment of the sieves for dumping and brushing and yet has enough flexibility to allow for rotary shaking and tapping. The picture to the right shows the sieves mounted on the belt. The sieves are clamped into a frame for the shaking phase of the analysis. This method of sieve attachment allows the Gradex 2000 to supply the same rotary shaking/tapping action as the more traditional RoTap shaker.



After shaking, each sieve is dumped then mechanically brushed. The contents of each sieve are then weighed and the data is sent to a PC. Using the weight data, the fractional and cumulative percentages retained on each sieve are calculated as shown below.





The fractional % retained and the cumulative % retained results are then printed and imported into the daily lab sheets or the sugar shipment program for generation of the certificate of analysis. Below is an example of the input sheet into which the percent cumulative data is imported with the calculation of the MA and CV for a strike sample.

White Sugar Input Data Sheet

Save Records and EXIT | Back | 24 Hours Ending @ 7am on 2/1/2007 | Ahead | EXIT And Don't Save

Today's Date is: Tuesday, February 13, 2007

#		Comp	11pm	11:30pm	12am	12:30am	1am	1:30am	2am	2:30am
1	WHITE PAN NUMBER				6816	6817	6818	6819	6820	
2	SUGAR PROD % TRANSMITTANCE	77.0	74.2		74.2	75.6	76.8	76.8	75.4	
3	SUGAR PROD COLOR	37	42		42	40	37	37	40	
4	SUGAR PROD CONDUCTANCE	19.3	15.1		14.1	14.0	14.0	14.3	20.7	
5	WATER CONDUCTANCE	0.7	0.7		0.7	0.7	0.7	0.7	0.7	
6	SUGAR PROD ASH	0.011	0.009		0.009	0.008	0.008	0.008	0.012	
7	SUGAR PROD SEDIMENT WHITE	2.0			1.0	1.0	1.0	1.0	1.0	
8	SUGAR PROD SEDIMENT BLACK	1.5			1.0	1.0	1.0	1.0	1.0	
9	SUGAR PROD MOISTURE	0.023			0.026	0.030	0.024	0.023	0.033	
10	SUGAR PROD SO2 ml. IODINE	1.2			1.1					
11	SUGAR PROD BLACK ml. IODINE	0.5			0.5					
12	SUGAR PROD IODINE FACTOR	3.92			3.92					
13	SUGAR PROD SULFITES - SO2 ppm	2.7			2.4					
14	SUGAR as PRODUCED TEMP						22			
15	[illegible]	0.00				0.00	0.00	0.64	0.00	
16	SUGAR PROD CUMM %-SCREEN #30	1.86				2.28	1.75	2.09	3.00	
17	SUGAR PROD CUMM %-SCREEN #35	10.75				17.48	11.55	18.43	13.83	
18	SUGAR PROD CUMM %-SCREEN #40	26.30				39.01	27.59	37.51	30.70	
19	SUGAR PROD CUMM %-SCREEN #50	69.00				77.36	67.95	74.22	70.39	
20	SUGAR PROD CUMM %-SCREEN #60	81.88				87.02	81.50	84.76	82.56	
21	SUGAR PROD CUMM %-SCREEN #70	88.74				91.96	88.76	91.06	89.02	
22	SUGAR PROD CUMM %-SCREEN #80	93.14				95.04	93.36	94.79	93.19	
23	SUGAR PROD CUMM %-SCREEN #100	96.29				97.40	96.49	97.41	96.29	
24	SUGAR PROD PASS SCREEN #100	3.71				2.60	3.51	2.59	3.71	
25	SUGAR PROD MA	0.0140				0.0152	0.0140	0.0151	0.0144	
26	SUGAR PROD CV	34				32	34	34	35	

Below is an example of Gradex results for a shipment Lot which has been imported into the sugar shipment database for use in the generation of the certificated of analysis.

Package Quality Analysis - New

PACKAGE | BULK | LIQUID

Product: 50= Fine Gran Sugar

M Start Date: 02/21/07 RESET

Lot Number: S052H

Analysis

Value out of range if text is red.

Gradex ID: TEST A

Screens	Cumulative	Fractional	PASS
20	0.1	0.1	99.9
30	3.1	3.0	96.9
35	16.1	13.0	83.9
40	40.6	24.5	59.4
50	76.2	35.6	23.8
60	87.2	11.0	12.8
70	92.2	5.0	7.8
80	95.8	3.6	4.2
100	97.5	1.7	
Pass 100	2.5	2.5	
MA	0.015		
CV	32		

Ash %	0.009
Color	34.
Moisture %	.023
Sediment PPM	0.
Visual Specks #/500G	0.

This is sub lot analysis number 1.

Save | Exit

Benefits of sieve analysis using the Gradex 2000:

The use of the Gradex 2000 allows us to automate this procedure while still using a method based on classic sieve particle size analysis. The software allows for the use of approximate sample weights for analysis with automatic calculation of % retained. This allows for simpler sample preparation especially when using riffled samples. The Gradex 2000 has consistent dumping and brushing with improved reproducibility when compared with manual weighing of samples. Using the Automatic Sample Feeder allows the analyst to set up and run up to seven samples unattended. This automation has allowed for the increased frequency of analysis of various sugar samples such as production sugar from each white pan several times a day using the results from a nine sieve screen test to determine the MA and the CV. Such an increase in frequency and number of sieves is also a safety issue when considering the potential for ailments arising from the repetitive motion involved in manually brushing out a set of nine sieves by an analyst. Comparison to data from the Rotap has been very consistent with a small increase in the amount captured on the US 30 and US 40 screens, giving a slightly higher MA. One interesting note is the same samples were analyzed on a vibratory shaker. These results gave a higher MA than both the Rotap and the Gradex with larger fractions retained on the larger sieves.

Averages of 10 Samples

	RoTap	Gradex	Gradex	Shaker	Ankersmid
MA	0.0135	0.0143	0.0142	0.0149	**0.0143**
CV	34.1	33.0	33.1	32.6	36.2

Some challenges using the Gradex 2000:

The automation of the Gradex requires many moving parts which must be monitored, kept clean, and in adjustment for good operation and dependable results. In tens years of operation, learning and modification by Rotex Inc., most of the initial problems experienced have been eliminated. Weekly cleaning of the sieves and the rings which hold the sieves in place seems to be important for trouble free operation. Monitoring the performance of the various air cylinders used in the analyzer, for leaks, is important for trouble free operation. Frequency of cleaning of the analyzer can also vary depending on the amount of "fines" in the sugar samples. More dust requires more frequent cleaning. Cleaning frequency and even sample results can be affected by static electricity charged samples. Low moisture, production sugar fresh from the cooler/dryer or handling samples in certain plastic containers can develop a static electrical charge strong enough to prevent the sample from freely falling through the screen stack resulting in higher % retained on coarser sieves. This has been a problem a few times in the years of Gradex 2000 use.

Particle analysis

When sugar shipments and production reached a level that required separate analysts, an investigation of alternative methods for granulation was initiated. Recognizing the need for particle analysis in a variety of wet and dry samples over an extended size range necessitated an instrument with flexible application setup. Particle Size distribution

(PSD) using microscopic and video evaluation along with laser diffraction and laser Time of Transition were evaluated for their range of applications, ease of use, and reproducibility including comparisons to sieve data.

Particle size, shape, and distribution are important to many factory operations. Sugar end Pan Operations call for larger crystals and a narrow distribution. A wider distribution of crystals in the wall of sugar in the centrifugal becomes less porous and centrifugal wash becomes less efficient and effective. Longer cycle times and greater amounts of wash water required are the result. Conglomerates, with the inclusion of mother liquor, are also difficult to wash and will contribute to higher color and ash.

In storage and handling particle size, shape, and distribution affect the ability of the material to flow. Conglomerates will break apart easily exposing surfaces which may release trapped moisture. Conglomerates breaking up, along with micro-particles, can cause dust hazards, including explosive potential.

Customers want a product that will flow freely out of storage without lumps. Particle size, shape, and distribution, along with other factors such as moisture migration, have a large effect on flowability. All else being equal, larger particles will flow with less force applied than smaller particles, as will particles approaching spherical shape as opposed to rods or other shapes. In the case of the centrifugal washing, a narrow distribution of particle size was desirable, but from a flowability standpoint a mixed distribution will flow more easily. The smaller particles get in between the larger ones and allow them to pass by each other with less friction.

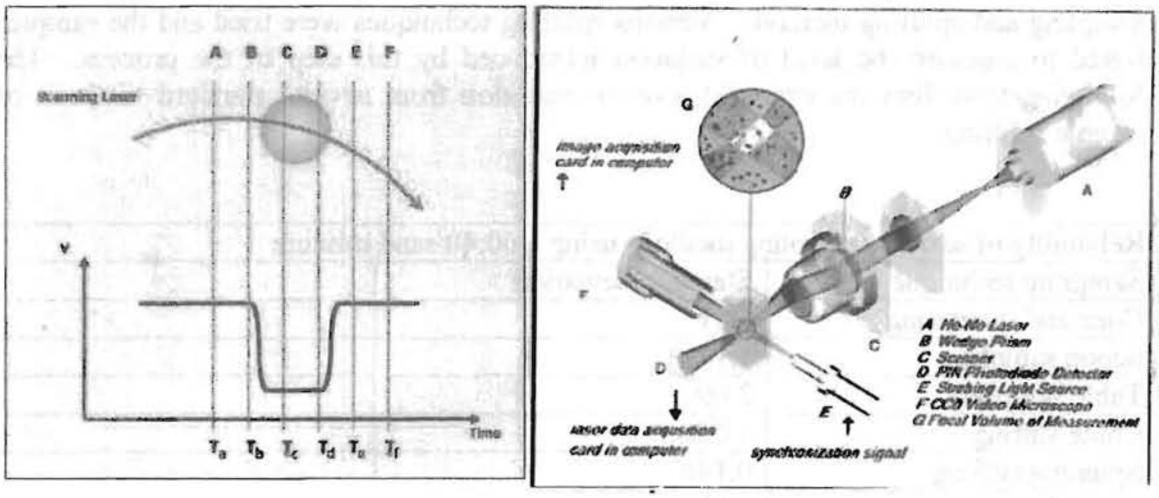
Particle size and shape can also have an effect on our customer's process. One example is a powder mix used to make a flavored drink. If fine particles increase too much, a lower density of the loose powder may result. If packaged by weight, the product may no longer fit in the packaging. Large particles will not dissolve as fast in water and fine powders may float on top or form clumps, both of which are difficult to dissolve.

Ankersmid CIS-100

Laser diffraction, laser Time-of-Transition, and video image analysis were the technologies demonstrated. Laser diffraction uses a system with a laser, lenses, and multiple detectors or detector zones. The theories used are the Fraunhofer and Mie theories. When the laser beam passes thru the particle zone the beam is diffracted by particles and the detectors produce a pattern which is interpreted by the Fraunhofer or Mie theories. This type of analysis assumes spherical particles. Because of this assumption it is difficult to correlate with sieve fraction data for non-spherical shaped particles like sugar.

The ability to perform particle analysis on dry or liquid suspensions from submicron to millimeters was very attractive. A number of different manufacturers were asked to demonstrate their particle size analyzers. Based on features, performance and price we purchased the Ankersmid CIS-100.

Laser Time-of-Transition is one of the analysis forms used by the Ankersmid unit we purchased. It is much more straightforward. A laser beam is passed thru a wedge prism rotating at a fixed rate (200 Hz) creating movement of the laser beam in a circular path. Samples are place in the laser path and a photodiode detector is placed at the end opposite the laser. Gravity fed or liquid suspended particles are detected by interruption of the laser/photodiode signal. Since the laser is moving at fixed rate, the time that the laser is obscured is used to determine the size of the particle. The shape of the signal Transition is used to reject particles encountered out-of-focus or off-center. If the sample particles are transparent or translucent the analysis can be setup to handle the signal differently as well. The use of Time of Transition makes the analysis independent of the particles' shape or its real or imaginary refractive index that is generally required for Laser Diffraction. We have used the laser ToT for analysis of liquid samples including carbonation, centrifugal wash water, and wastewater samples. This method suffers from the same shortcoming of assuming spherical particles.



Diagrams from Ankersmid B.V.

Production sugar is analyzed using the video channel. Images are acquired in real-time and can be analyzed for many size and shape parameters. One parameter that may be of interest is the shape factor. This is indicative of how smooth or rough the sample particles are and may indicate the presence of agglomerations. Another shape parameter is the Aspect ratio. This parameter indicates the ratio of a particle's width to its length. A higher aspect ratio indicates a spherical particle, while a lower ratio indicates a rod shape. This Transition in particle shape may be indicative of the presence of crystal habit modifiers like raffinose or dextrans. The variation of the sample's aspect ratio can yield interesting difference in the size distribution results of sieve analysis versus laser or video imaging techniques. Particle analysis with sieves uses a two dimensional aperture to determine the size of a three dimensional particle. Particles will pass through a sieve based on its 2nd minimum diameter. The probability of a rod shaped particle passing through a screen depends on its length in addition to the motion and force applied to the screen set. The gyratory motion combined with a tapping force of standard methods will help fluidize the bed of sugar particles and allow them to transition to the vertical position needed for passage. Factors such as the size of the charge load change the depth of the particles on a screen and can affect the efficiency of fluidization.

Laser and video image analysis also take a two dimensional look at the sugar particle. As the sugar moves across the detector it leaves a shadow that is captured for analysis. This difference will be seen as the orientation of the particles' axis will vary and the particle size will be seen as an averaged result. By using the video mode, the CIS-100 can be set to analyze based on the Minimum Ferrets or diameter. The use of the minimum ferret parameter allows us to come close to the second smallest diameter found in sieve analysis.

Sampling methods

The initial testing of the Ankersmid was done with duplicate samples using a chute type sample riffler. The variation of the initial results was disappointing. While repeated analysis on the same sample was very reproducible, the comparison between hand sieve and the video analysis had a high level of variation. One step was to improve our sampling and splitting methods. Various splitting techniques were tried and the samples tested to measure the level of variation introduced by this step of the process. The following table lists the expected level of variation from several standard methods of sample splitting.

Reliability of selected sampling methods using a 60:40 sand mixture		
Sampling technique	Standard deviation	
Cone and quartering	6.81	
Scoop sampling	5.14	
Table sampling	2.09	
Chute slitting	1.01	
Spinning riffling	0.146	
Random variation	0.075	

Table from Table 1.5 page 38, T.Allen Particle Size Measurement, 5[th] Edition Volume 1 Chapman and Hall 1997 ISBN 0 412 72950 4

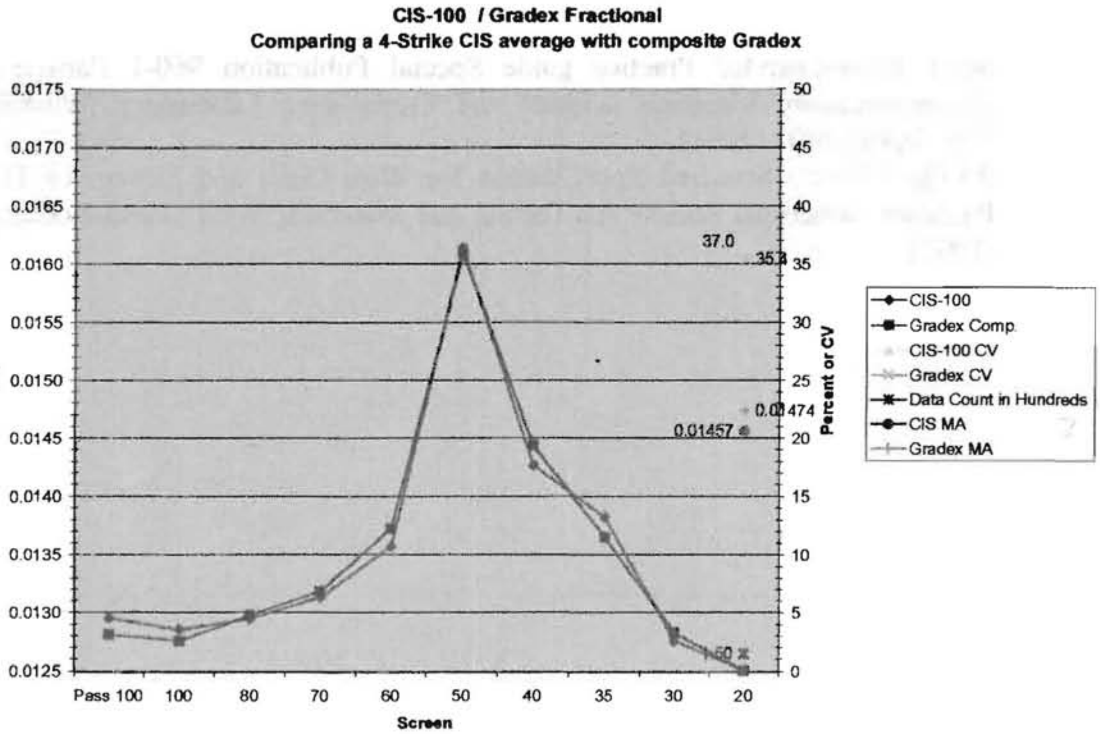
The solution to variation in sample splitting was to run the same sample on both the video instrument and the Gradex. When analysis for production sugar was run on the Ankersmid unit we would collect a composite of four runs. This composite was then analyzed on the Gradex for comparison with the average of the four from the Ankersmid analyzer.

The evaluation of MA and CV data derived from sieve and video analysis has been satisfactory and yielded good results even with the differences in handling of the crystal's physical differences.

One addition is the ability to provide particle distributions based on volumes, which is used to compare to sieve analysis, but also on numbers. This distribution provides a significantly different outcome. The impact of the large number of fine particles in a given volume or weight of a sample is clearly evident. A particle size distribution by numbers will give a double peaked graph showing the large number of particles smaller than 150 microns.

One measurement of concern was the results on individual screens. Granulation control for optimizing operations is important but is normally based on MA and CV data. In addition the coarse (US 30) and fine screen (Pass 100) data is also important to meeting many customer specifications. In this instance matching data between the two methods was difficult to correlate, especially for the sub 150 micron particles of the pass 100 screen. Samples of the fractions caught on individual screens were analyzed on the CIS-100. These samples often showed residual particles that did not pass through the sieves, and was collected on coarser screens but which were found using video analysis.

Overall the data provided matched well when averaged, but determining the results for individual ranges was effected by how the instruments handled the rod shape of the sugar crystal and the finer particles. In order to provide specific screen data, the video and sieve analysis were used to develop a correction factor to reliably predict actual sieve results.



The CIS 100 was also purchased to provide additional data on particle size in process streams. The instrument can be equipped with several different accessories which allow for the analysis of liquids, solids, and viscous materials. We have completed some preliminary work on samples from fondant preparation, carbonation, and water treatment. Fondant samples were prepared using vibratory mills and rod mills using different grinding media and grind times. The vibratory mills run at 60 hz and are able to grind the sugar and isopropanol mixture to a 10 micron average size within 15 minutes. However, analysis showed with longer grinding times the variation of the particles was improved.

It appeared the mills reached a minimum size and with longer grinding the larger particles were reduced closer to the average size. Overall the least amount of variation was found with the vibratory mills at about 6 hours of grinding.

Particle analysis is one of the main ways of determining sugar quality. The search to automate this method requires a good understanding of the underlying principles that affect the quality of the data obtained. The Gradex 2000 sieve analyzer has proven to be a reliable instrument which provides consistent results and helps protect the health of our analysts. The addition of the Ankersmid CIS-100 has given us the ability to analyze a wide range of dry powders and liquid samples. We plan to examine additional attachements for the CIS 100 which may allow us to analyze viscous samples like Pan mass. Both instruments have been important additions to our process control and quality programs and will provide new insights into the processing of sugarbeets .

SMBSC would like to thank Upasiri Samaraweera of Minn-Dak Growers' Cooperative and Vince Salzman of Wyoming Sugar for sharing data and providing samples to analyze and compare.

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