

Observations in Low Raw Crystallization

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Introduction

The development of operating guidelines through trial and error often bypasses optimum conditions. Such a compromise may have substantial economical shortcomings. In low-raw crystallization these shortcomings become evident through below potential extraction, an inefficient utilization of available facilities, and an unnecessary increase of recycled nonsugars which burdens the entire sugar house.

Methods and techniques now are available which allow an accurate evaluation of operational weaknesses in the system.

Methods and Development

A previously published paper (1)² reported on the techniques employed to evaluate low-raw crystallization. As a continuation of this program the findings of the previous investigation were applied to actual plant operation.

During the 1964-65 campaign, all five factories of The Amalgamated Sugar Company were systematically investigated with regard to their performance on the low-raw side. The basic program involved:

- A. Withdrawal of massecuite at pan drop and at various intervals from the crystallizer.
- B. Separation of molasses from its respective sugar crystals.
- C. Determination of specific solubility and viscosity characteristics.
- D. Keeping of an accurate log for all pertinent plant operating data.

All values for percent sugar, nonsugar and derivations thereof are based upon refractometer dry substance and Clerget acid true purity determination.

Results and Discussion

The solubility of sucrose in molasses from the 1964-65 campaign is illustrated for all five factories in Figure 1. Potential molasses purities calculated from these solubilities are shown in Table 1.

The change in sucrose solubility at Minni-Cassia, illustrated by a drop in purity potential, within a matter of a few weeks indicates the effects of a possible change in the nonsugars.

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² Numbers in parentheses refer to literature cited.

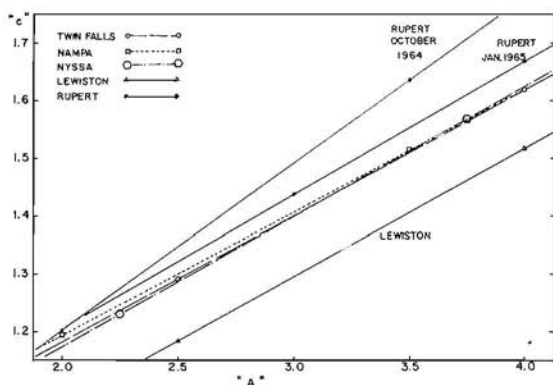


Figure 1.—Relationship between the coefficient of saturation “c” and the nonsugar/water ratio “A” during the 64/65 campaign. All factories.

Table 1.—Potential molasses purities.

NS/W or A	Purging temperature 55°C	
	2.75	2.5
Lewiston	55.16	56.39
Nampa	57.34	58.68
Twin Falls	57.18	58.49
Nyssa	57.12	58.40
Minni-Cassia, Oct/Nov 1964	58.47	59.52
Minni-Cassia, January 1965	57.77	59.05

Variations in solubility are dictated by the respective nonsugar compositions. With exceptions the most pronounced influence upon sucrose solubility was attributed to variations in the alkali and raffinose concentrations, respectively. Sucrose solubility increased with rising levels of potassium and sodium and decreased, respectively, with rising levels of raffinose. Some data on nonsugar composition for the material involved in this program are shown in Table 2.

Table 2.—Nonsugar constituents in sugar beet liquors—campaign 1964-65¹.

Factory	Lewiston		Minni-Cassia	Twin Falls	Nampa	Nyssa	
	1964-65 Campaign	Oct. 1964	Nov. 1964				January 1965
Kjeld, Nitrogen		4.86	5.62	5.45	5.12	6.32	5.66
Ash		24.67	31.79		28.56	25.7	26.64
Raffinose		10.15	2.76	4.31	6.25	4.22	5.12
Sodium		2.36	2.02	2.93	3.04	2.42	3.28
Potassium		10.47	14.26	12.35	11.54	11.03	9.83
Sod + Pot eq/100 NS.		0.37	0.45	0.44	0.43	0.39	0.40

¹ All values as percent of total nonsugars. Total nonsugars as calculated by refractometer dry substance and Clerget single acid true purity. Raffinose by Clerget single acid TP.

Proper use of the determined solubilities permits the accurate assessment of supersaturation at any given time. This allows the optimum scheduling of cooling rates and water dilution within the range of acceptable viscosities during the crystallizer cycle. The operation at two plants was of particular interest and shall be discussed in this paper.

Table 3 illustrates the low raw pan performance for a Steffen factory. The values represent the averages from eighteen strikes and are considered typical.

Table 3.—Low raw pan performance. Comparison at pan drop.

Data are averages of six strikes from each pan			
Pan number	1	2	3
Mechanical circulator	No	Yes	Yes
Massecuite RDS	95.08	95.81	96.24
Massecuite purity	79.71	80.24	79.52
Massecuite temperature	95.2	94.1	94.4
Molasses purity	67.01	64.63	64.48
Nonsugar/water ratio	3.96	4.56	5.24
Molasses supersaturation	1.11	1.1	1.14
% sugar crystallized	47.9	54.4	53.25

The large daily work-load of this particular plant allowed a turn around time of less than four hours for each of its three low raw pans. It is of interest to note the inferior performance of pan 1. In spite of higher massecuite temperatures pan 1 could not be brixed as high as the other two pans. This trend also is reflected in a higher molasses purity at pan drop and a lower percentage of crystallized sugar. One pan with an abnormally high massecuite purity somewhat distorted the average for pan 2 with respect to the nonsugar/water ratio at pan drop. The averages for pan 2 without the values from the one abnormal pan were nearly identical to the values for pan 3. Attention is called to the relatively uniform supersaturation which must be ruled out as the reason for the inferior performance of pan 1.

The extremely short boiling time of only about 3.5 hours was possible because of high crystallization rates at the high temperatures. Supersaturations in excess of 1.15 have never been observed during this study in low raw massecuite at temperatures around 95°C.

However, the high pan drop temperatures make it necessary to increase the crystallizer cooling rate in order to maintain the schedule. With six rotary Lafeuille type crystallizers and a subsequent cycle time of less than eight hours, the potential lowest molasses purity could not be reached. A typical crystallizer cycle is illustrated in Figure 2.

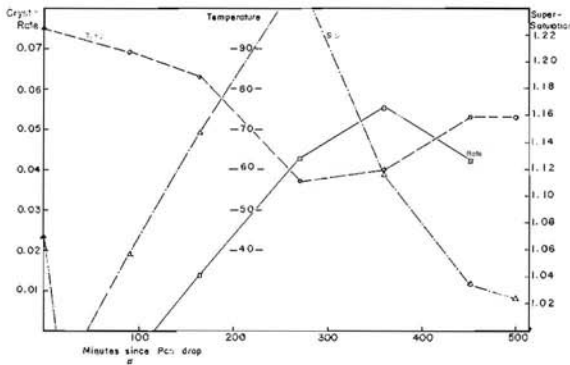


Figure 2.—Relationship between temperature, supersaturation and cumulative rate of crystallization in mother liquor during a complete crystallizer cycle. Nyssa factory 1964-65. Low raw pan no. 2, Lafeuille crystallizer no. 5, Pan boiling no. 802.

It was common practice at this plant to add some dilution water and maintain pan drop temperature respectively at the start of the crystallizer cycle. Such practice was predicated on the assumption of dangerously-high supersaturation at the final brixing stage. The evidence presented challenges the validity of such assumptions. In each case the supersaturation was reduced through this practice at the beginning of the crystallizer cycle to a level which virtually stopped crystallization. Variations in massecuite brix and purity at pan drop under conditions of uniform crystallizer treatment produced an undersaturated condition early in the cycle for about $\frac{1}{3}$ of all crystallizers tested.

Only after an adequate build-up of supersaturation through progressive cooling could crystallization resume. This is indicated by the rate of crystallization as illustrated in Figure 2.

$$\text{Rate} = \frac{100 (S - S_1)}{S T}$$

where: T = Minutes since pan drop.

S = Dissolved sugar at pan drop.

S_1 = Dissolved sugar after T minutes.

In order to meet the allotted crystallizer turn around time, the operators had to resort to extremely fast cooling rates later in the cycle thereby forcing an excessive build-up of supersaturation at a rather inopportune time when increasing viscosity begins to become a significant operating problem. This is the period when the danger of additional nucleation is greatest, thus, threatening serious deterioration of purging quality. The usual remedies to which operators resort are either high purging temperature

and/or addition of copious amounts of water. Summarized crystallizer performance is shown in Table 4.

Table 4.—Crystallizer performance. Comparison at discharge.

Masseccite from pan no.	1	2	3
Masseccite temperature	68.2	68.9	64
Nonsugar/water ratio	2.92	3.17	3.53
Molasses purity	61.15	59.68	58.54
Molasses supersaturation	1.04	1.02	1.03
% sugar crystallized in crystallizer	11.23	9.16	10.38
Rate of crystallization	0.048	0.043	0.049

On the basis of these findings the operators were advised to drop temperatures faster at the beginning of the crystallizer cycle in order to take advantage of the more rapid crystal growth at the high temperatures. More time was thereby made available to cool more gradually in the lower temperature range where crystal growth is substantially impaired. The danger of additional nucleation during the crystallizer cycle was thus greatly reduced as verified by improved purging operation and reduced molasses purity.

The explorations at another plant, a straight house, were designed to provide effective control parameters for the operation of recently-installed continuous Stearns-Roger crystallizers. Figure 3 shows a schematic control diagram for a continuous crystallizer which was designed in cooperation with the Taylor Instrument Company.

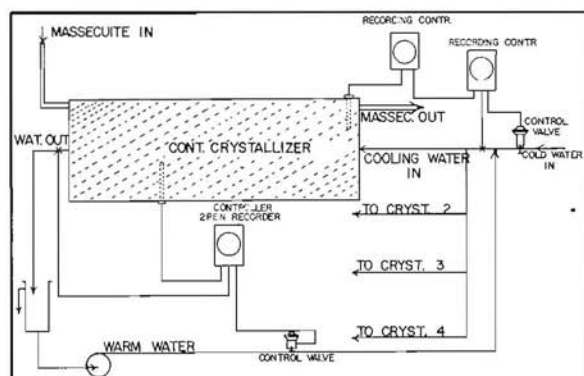


Figure 3.—Schematic diagram of temperature control system for continuous crystallizer.

The main objective of this control system is to keep the ΔT between the cooling water and the masseccite as small as possible while achieving progressive cooling of masseccite toward the discharge end. The system was installed on an experimental basis on one crystallizer only and led to its application

at the other three units. A common control of cooling water temperature for a number of crystallizers may be achieved as outlined in Figure 3. However, volume control for cooling water through each respective unit should be handled separately, allowing adjustments for variations in ambient temperature and/or massecuite flow.

A schematic flow diagram shown in Figure 4 illustrates the general layout of the crystallizer station.

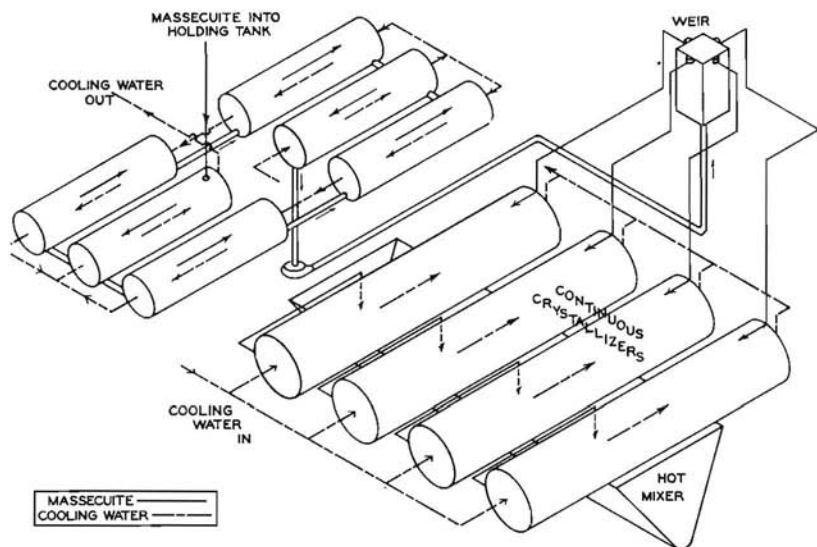


Figure 4.—Schematic flow diagram for low raw massecuite and cooling water.

The original arrangement provided for receiving pan massecuite in either of the two center holding tanks and using the two tanks to each side of these holding tanks as overflow reservoirs through bottom connections. Massecuite was withdrawn from bottom outlets of center tanks and pumped to the Weir overflow for distribution to the continuous crystallizers.

Raw pans were dropped alternately into either of the two independent holding tanks. Pan drop temperature in the holding tanks was maintained by circulating hot water through the existing cooling coils. The temperature fluctuations of massecuite entering the continuous crystallizers was thus minimized.

Water addition to the crystallizers at the beginning of the 1964-65 campaign was programmed according to the solubility data from 1963-64. A molasses single acid true purity of 58.5% was expected at a final nonsugar/water ratio of 2.5 and at a

Table 5.—Performance of the low raw side.

October/November 1964		
	Pan	Crystallizer
Massecuite discharge from		
No. of strikes	13	15
Massecuite RDS	93.48	91.71
Massecuite purity	77.92	
Massecuite temperature	78	46.3
Molasses purity	66.07	59.75
Nonsugar/water ratio	3.277	2.395
Supersaturation	1.1056	1.096
% of sugar crystallized	44.67	59.44
	Pan drop	Crystallizer drop

purging temperature of 55°C. Table 5 represents the values actually obtained.

Solubility tests with the 1964-65 campaign material uncovered the shift in sucrose solubility as illustrated in Figure 1.

By the end of November the change in the flow pattern through the holding tanks to the outline as shown in Figure 4 was completed. The new target nonsugar/water ratio at purging was raised to 2.75 to account for the increased sucrose solubility found for the 1964-65 nonsugar media. Results obtained under the changed conditions are shown in Table 6.

Table 6.—Low raw fillmass characteristics after modification of flow scheme.

January 1964-65		
	Pan	Crystallizer
Massecuite discharge from		
No. of strikes	13	
Massecuite RDS	93.83	92.89
Massecuite purity	78.46	
Massecuite temperature	80.3	44.2
Molasses purity	67.32	58.33
Nonsugar/water ratio	3.281	2.811
Supersaturation	1.214	1.161
% of sugar crystallized	43.47	61.67

The progressive cooling achieved according to Figure 4 by interconnecting all six holding tanks and subsequent counter-current flow of massecuite and water respectively shifted the main crystallization after pan drop to the holding tanks where temperatures above 60°C prevail. Time now was available to allow reduced cooling rates through the continuous crystallizers during the low temperature range where viscosity is at maximum and crystal growth at minimum in spite of relatively high supersaturations.

The above changes together with an improved pan boiling procedure brought remarkable improvements in purging quality. Although the adjustments on the pan floor cannot be credited with a lower molasses purity, very uniform crystals conducive to

good purging resulted. Observations in low raw crystallization repeatedly confirmed that excellent crystallizer operation cannot overcome bad boiling practices. By the same token poor crystallizer treatment can easily ruin the best strike. Table 7 gives a direct comparison of crystallizer achievements before and after the indicated changes.

Table 7.—Sugar crystallized through crystallizer station as percent of total sugar in massecuite.

	Holding tank	Continuous crystallizer	Total crystallizer station
Before changes	6.52	8.25	14.77
After changes	12.59	5.61	18.2

Table 8 illustrates the progressive molasses exhaustion through the entire low raw end. The four sample cocks on the continuous crystallizer divided the continuous crystallizer essentially into five compartments of equal size. Optimum performance is evident throughout the crystallizer station with exception of the mixer above the centrifuges.

Table 8.—Progressive crystallization through the low raw end—Minni-Cassia, January 1965.

	% sugar crystal	Increase	NS/W	S.S	Temp.
Pan drop	43.47		3.27	1.214	80.3
Weir	56.06	12.59	3.166	1.096	66.8
Sample cock 1	58.06	2.00	3.273	1.186	58.5
Sample cock 2	59.22	0.76	3.267	1.213	53.4
Sample cock 3	60.69	0.47	3.267	1.215	49.7
Sample cock 4	61.05	0.36	3.004	1.206	46.3
Discharge	61.6	0.55	2.812	1.161	44.1
Gooseneck cold*	62.17	0.57	2.872	1.196	39.8
Gooseneck hot	57.81	-3.79	2.892	1.08	60.1

* Only one half of the mingler above the centrifugals was heated.

One section of the low raw mixer was equipped with Stevens coils during this period. Excessive heating of massecuite in the hot section of the raw mixer was responsible for a general purity increase of molasses through the raw mixer. Raw mixer stations in all factories exhibited some weaknesses resulting either in purity increases and/or inferior purging quality respectively. The objective of any well-controlled hot mingler should be to hold molasses purity while reheating the massecuite to the optimum purging temperature. This objective was not achieved. The records show that poor heat transfer caused some localized overheating resulting in dissolving some sucrose. The ideal conditions are to reheat the fillmass to a purging temperature just short of

reducing the supersaturation below 1.0. Other disadvantages compound the problem such as extra long gooseneck sections which hold a major portion of a centrifuge charge and excessive variations of ambient temperature where mingler and goosenecks are not insulated from cold drafts.

However, in spite of these problems it is essential for maximum molasses exhaustion to optimize the raw mixer operation especially for those equipped with a hot coil. A nomograph as shown in Figure 5 (1)² was a valuable tool for the selection of correct purging temperatures.

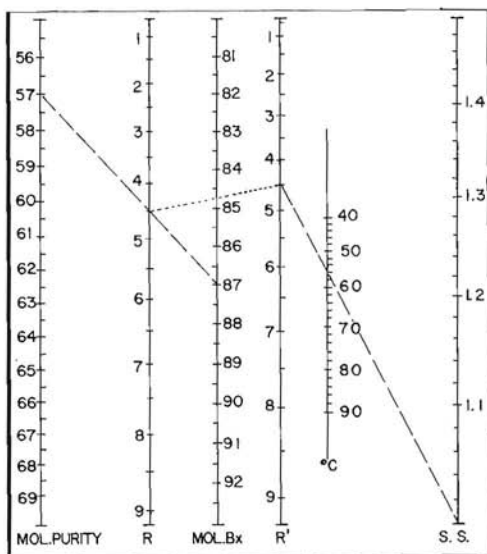


Figure 5.—Nomograph showing relationship between purity, brix, temperature and supersaturation.

The mathematical relationship as expressed graphically in Figure 5 may be illustrated thusly:

$$SS = \frac{S}{S'} = \frac{ZK'}{100}$$

where: SS = Supersaturation

S = Total dissolved sugar in grams/100 grams of water at temperature "T"

S' = Grams sugar/100 grams of water at saturation and temperature "T"

Z = Grams sugar/100 grams of water at saturation and temperature "T" for pure sucrose solution.

² Numbers in parentheses refer to literature cited.

$$K' = F \frac{(B)}{(P)} = \frac{N + \frac{N'}{B}}{P} - m.$$

$$K = K' + m$$

B = Refractometer dry substance

P = True purity of solution (Clerget)

N = A constant = $100 m - 100 b = 52.5$

N' = A constant = $10^4 b = 7450$

m = 0.217 (slope of solubility curve)

b = 0.745 (Y — intercept of solubility curve)

Values for R and R' in Figure 5 are substitutions for K and K' respectively to simplify the use of the nomograph. Figure 5 was applicable for low raw massecuite material from the Nyssa, Nampa and Twin Falls areas during the 64/65 campaign. A change in the solubility will yield different constants and may subsequently change the relationship within the nomograph.

All values given in Table 8 are calculated averages and include a large number of samples from each of the three crystallizers in operation at that time. Differences between crystallizers were small. Variations between samples taken at the same location but at different operating periods were also small. Dilution water was added as close to the discharge end of the continuous

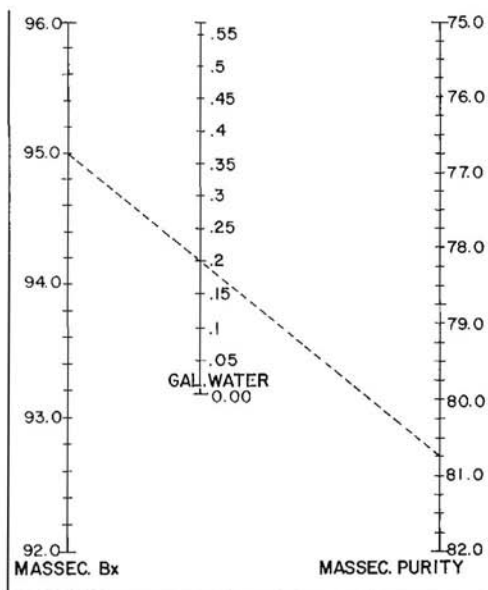


Figure 6.—Water addition to 100 lbs massecuite for NS/W of 2.75 at purging.

crystallizer as possible and still have complete dissipation before massecuite discharge.

A nomograph as shown in Figure 6 was used by the operators to schedule water addition to the massecuite.

Values obtained from this nomograph were multiplied by a factor representing a function of massecuite pump stroke and displacement, respectively. The product of this multiplication was in gallons of water per minute required to yield a final nonsugar/water ratio of 2.75.

$$\text{Thusly: } W = \frac{(^{\circ}\text{Brix} \times P_1) - 100}{8.33}$$

Where:

W = Water addition in gallons/100 lb massecuite

$^{\circ}\text{Brix}$ = Refractometer dry substance

$$P_1 = \frac{(100 - P) + 275}{275}$$

P = % sugar

Figure 6 is a graphic expression of above formula.

Summary

The development of precise operating guidelines has become an extremely beneficial tool for optimizing low raw crystallization. However, it was found necessary to recalibrate these guidelines through occasional sucrose solubility tests. A shift in specific solubility for sucrose in molasses indicates a change in the alkali and/or raffinose concentrations, respectively.

Literature Cited

- (1) SCHOENROCK, K. W. R. and J. R. JOHNSON. 1966. Recent advances in molasses exhaustion. *J. Am. Soc. Sugar Beet Technol.* 13 (8): 662-680.
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