Mathematical Approach to Determine Vacuum Pan Boiling Time for Full Seeding Method Using Milled Fondant

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Sucrose crystallization rate is a controlling factor in sugar end operation. There are several methods available at the present time for determining sucrose crystallization rate in the laboratory. In fact many methods, proved to be satisfactory in the laboratory, have failed to be applicable in actual full-scale operation. This is particularly true in pan boiling.

The purpose of this investigation was to develop a suitable equation for use at the factory level for determination of pan boiling time.

Equipment

The following equipment was used in this investigation:

(1). White calandria pan.
3,340 sq.ft. heating surface, 2¾″ OD, 1677 bi-metal tubes, 0.065″ steel outside, 0.042″ copper inside, 3′—⅜″ long, 437 cu.ft. graining volume, 1750 cu.ft. strike volume, 15′, 0″ OD pan diameter. 13′—6″ OD calandria diameter, 75 hp circulator at 75 RPM, steam at 10 PSIG.

(2). Pan automatic controls.
Pan instruments were supplied by Taylor Instrument Company.
The automatic controls consisted of the following items:
- (a) Consistency probe and controller.
- (b) Level controller and transmitter.
- (c) Temperature controller and transmitter.
- (d) Absolute pressure controller.

(3). George Lory design, Lasico crystalscope (pan microscope) was inserted into the pan just below the normal graining level. Crystalscope micrometer has a scale which is 5 units long. Each unit is divided into 20 subdivisions. Each subdivision is equivalent to 0.001 inches. Total length of scale is 0.1 inches.

(4). Polaroid camera.

(5). Complete ball mill to make the fondant.

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Equations

Rate of decrease in mother liquor purity is determined by the degree of super-saturation and purity; namely

$$-\frac{dP}{dt} = k\ P\ (S\text{-}1) \qquad \ldots\ldots\ldots\ (1)$$

Degree of super-saturation is the ratio of the sugar in solution per 100 units water to the true solubility of sugar in that solution per 100 units of water both at the same temperature. Purity is the percentage ratio of sugar to total solids.

When degree of super-saturation of mother liquor is equal to 1, the seeded crystals will neither dissolve nor grow in the mother liquor. Therefore, the purity of liquor will not increase nor decrease with time. In this case the above equation becomes

$$\frac{d\ P}{d\ t} = 0$$

If purity of mother liquor is zero, sugar content of mother liquor is zero and will not change with time. Therefore,

$$\frac{dP}{dt} = 0.$$

Rearranging Equation (1) to $\frac{dP}{P} = -\ k\ (S\text{-}1)\ dt$ ..... (2)

Integrate Equation (2) between $t = 0$, $P = P^\circ$ and $t = t$, $P = P$ at constant super-saturation S.

$$\ln P - \ln P^\circ = -\ k\ (\ S - 1\ )\ t$$

Hence, $\ln P^\circ - \ln P = +\ k\ (\ S - 1\ )\ t$ ..... (3)

Solve for ln P, $\ln P = \ln P^\circ - k\ (\ S - 1\ )\ t$ .... (4)

Solve for t, $t = \frac{\ln P^\circ - \ln P}{k\ (\ S - 1\ )}$ .... (5)

At $t = 0$, Equation (4) is reduced to $\ln P = \ln P^\circ$. Hence, $P = P^\circ$, purity of mother liquor at graining is equal to the purity of standard liquor.

If S approaches to infinite super-saturation; that is,

$$t = \lim_{S \to \infty} \frac{\ln P^\circ - \ln P}{k\ (\ S - 1\ )} = 0$$

Time required to boil the pan from initial purity P° to final purity P is zero.

If super-saturation of liquor approaches to 1,

$$t = \lim_{S \to 1} \frac{\ln P^\circ - \ln P}{k\ (\ S - 1\ )} = \infty$$

Time required to reduce the mother liquor purity from P° to P is infinite.

From theoretical and actual standpoint, it is desirable to boil the pan at higher super-saturation. However, super-saturation above 1.5 should be avoided for full seeding method, because super-saturation above 1.5 is in the Labile zone which will cause the formation of fine grain and unwanted grain size.

There is an optimum condition at which fine grain will not form and the boiling time is minimum. This condition exists at super-saturation of 1.5 which is the upper limit of Metastable zone. In this zone, the crystal will grow but practically no new crystal will form.

Equation (4) is to be used to estimate the final green purity or mother liquor purity P for boiling the pan from initial purity P° and constant super-saturation S for total boiling time t.

Equation (5) is to be used to estimate the boiling time for boiling the pan from initial purity P° to final green purity P at constant super-saturation S.

It is rather difficult to boil the pan at constant super-saturation without proper and reliable instrumentations. Fortunately, Taylor Instrument Company has developed a temperature measuring device which is quite unique in pan instrumentations. The temperature measuring element is called Transaire Temperature Capillary Bulb which was inserted in the vapor line between pan and condenser. This temperature sensing bulb is quite sensitive, measuring any small change in the temperature of super-heated vapor leaving the surface. This temperature change is amplified and recorded on a recorder calibrated for 20° C span over full chart. With this temperature-measuring instrument and proper recording chart shown on Figure 1 together with accurate constant super-saturation curves for fillmass as shown on Figures 2, 3 and 4 for 6″ Hg abs., 7″ Hg abs. and 8″ Hg abs. respectively, it is possible to boil the pan at constant super-saturation.

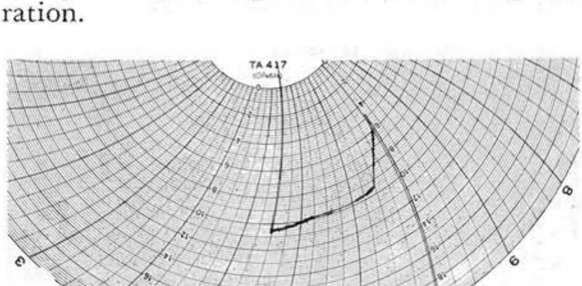

Figure 1.

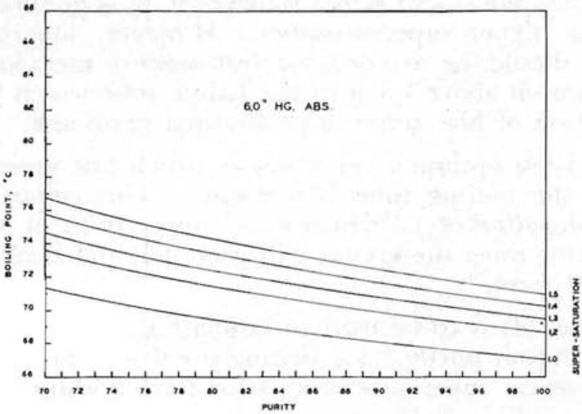

Figure 2.—Constant super-saturation lines.

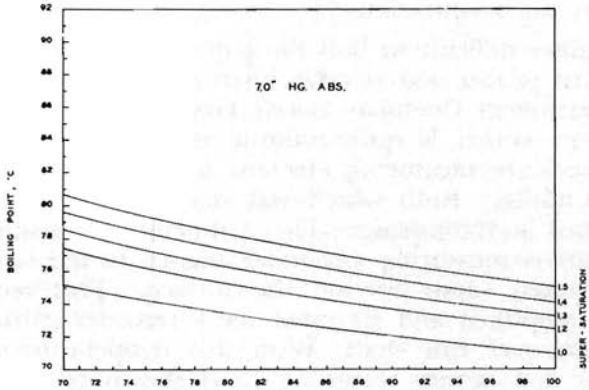

Figure 3.—Constant super-saturation lines.

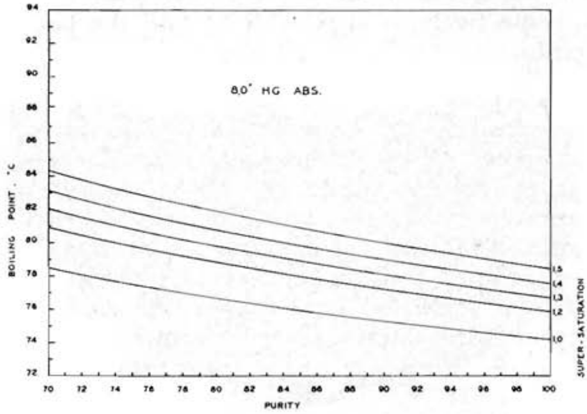

Figure 4.—Constant super-saturation lines.

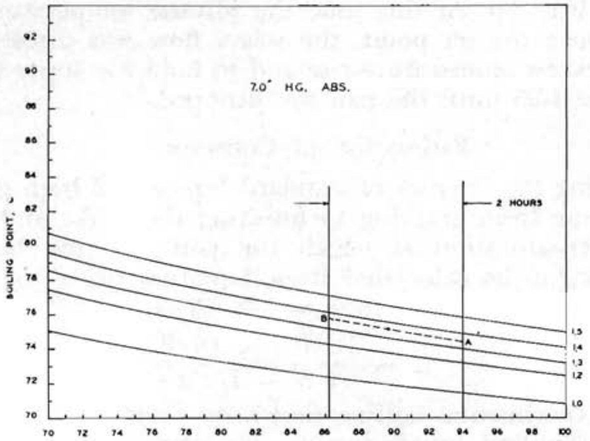

Figure 5.—Typical pan boiling at constant super-saturation.

On Figure 5, line AB shows the typical boiling cycle. Point A indicates the initial juice purity and temperature at graining. Point B indicates the final mother liquor or green purity and temperature. Line AB is on the constant super-saturation of 1.35. During boiling period AB, sugar from the mother liquor is depositing on the surface of existing sugar crystals at very rapid rate. Therefore, the purity of mother liquor is decreasing with time although the pan is still feeding with fresh high purity standard liquor. Hence, raising the fillmass temperature from point A to point B will not raise the super-saturation provided that absolute pressure of the pan remained the same.

Boiling a Strike

The strike was initiated by turning a switch which opened feed and condenser valves. When juice level reached the graining volume, steam was turned on to concentrate the liquor and the level maintained constant. An alarm sounded when super-saturation reached to pre-set value of 1.35. Then the pan was seeded with 200 c.c. of milled fondant. As the boiling proceeded, fillmass temperature tended to rise above the set point which was 1.35 super-saturation, the steam flow was throttled to prevent exceeding that limit. As the pan came together, the increasing tightness opened the feed valve. The combination of feed and increased crystal areas tended to cause the super-saturation to fall away from 1.35. Therefore, the steam valve was opened to maximum to keep the super-saturation constant at 1.35. Boiling proceeded until the pan reached the maximum set level, the feed valve was throttled to prevent further rise and the pan began to brix up. At this time the fillmass temperature started to rise above the set point, the steam flow was throttled again to avoid excess temperature rise and to hold the super-saturation constant at 1.35 until the pan was dropped.

Estimation of Constant K

Knowing the purities of standard liquor and high green, the boiling time from graining to finishing the strike and the constant super-saturation at which the pan is being boiled, the constant k can be calculated from Equation (3).

$$\ln P^{\circ} - \ln P = -k\,(S-1)\,t$$

Solve for k,
$$k = \frac{\ln P^{\circ} - \ln P}{(S-1)\,t} \quad \ldots\ldots\ldots (6)$$

For the experimental boiling shown on Figure 1:

initial standard liquor purity = 94.0
final high green purity = 86.2
super-saturation = 1.35
boiling time = 120 minutes
absolute pressure = 7″ Hg abs.

k can be estimated from Equation (6).

$$k = \frac{\ln 94.0 - \ln 86.2}{(1.35-1)\,120} = \frac{0.08662}{0.35\,(120)} = \frac{0.08662}{42} = 0.002062$$

During the boiling time of 120 minutes, temperature of fillmass was controlled so that super-saturation of fillmass not to exceed or drop below 1.35. This was very difficult to accomplish because the location of 1.35 super-saturation was not clearly known from the recording chart shown on Figure 1. Therefore, after the strike was completed, the temperature chart was examined for intermediate points to determine if the entire boiling was conducted at constant super-saturation of 1.35. This was done by computing the intermediate mother liquor purity P_i using the k value obtained above, time t after graining and constant super-saturation of 1.35. P_i was located on Figure 3 at 1.35 super-saturation and the corresponding fillmass temperature was read. The resulting fillmass temperature was the same as the temperature recorded on Figure 1. Hence, the pan was boiling at constant super-saturation of 1.35.

For the experimental boiling:
$k = 0.002062$, $P^{\circ} = 94.0$, $t = 30$ minutes after graining, super-saturation = 1.35.

Substituting the above data into Equation (4):

$$\begin{aligned}\ln P_i &= \ln 94 - 0.002062\,(1.35-1)\,30\\ &= 6.84588 - 0.002062\,(0.35)\,30\\ &= 6.84588 - 0.02165 = 6.82423\end{aligned}$$

Hence, $P_i = 91.9$ purity.

Fillmass temperature of 74.8° C was obtained by locating P_i of 91.9 on Figure 3. Fillmass temperature of 9.8 chart units, which was 74.8° C, was obtained by locating a point 30 minutes after graining on Figure 1. Both temperatures shown above were exactly the same, therefore, the pan was boiling at constant super-saturation of 1.35 from $t = 0$ min. to $t = 30$ min. The boiling curve was checked at 15 minute intervals in the same manner.

It was found that for boiling period of 2 hours and 1° C temperature rise between graining and dropping the pan, the entire boiling was conducted at constant super-saturation provided that the line between initial and final points was fairly straight and the absolute pressure remained constant.

To verify the value of k calculated from Equation (6), the following procedure was used: Using Polaroid camera, series of pictures were taken from pan microscope at 15, 20, 25, 40 and 110 minutes from graining as shown on Figures 6, 7, 8, 9 and 10. The volume of fillmass in the pan was recorded for each picture taken. With this data, together with the known quantity of milled fondant used, it was possible to calculate the constant k.

Milled fondant was prepared by adding one pound of granulated sugar with a liter (1000 c.c.) of iso-propyl alcohol in the mill jar. The milling was continued for exactly 24 hours. It was found that times of milling in excess of 24 hours didn't accomplish any finer grinding but times of less than 23 hours were found to produce inferior seed.

For milling time of 24 hours, 1 c.c. of fondant contains 2.5×10^9 crystals. The average crystal size has been observed to be 4.5 microns or O.000177″.

For experimental boiling 200 c.c. of milled fondant was used to grain the pan. Total number of crystals in 200 c.c. $\doteq 5 \times 10^{11}$ crystals. From Figure 6, average grain size was 0.006″ at 15 minutes after graining.

Volume of fillmass at 15 minutes after graining was 437 cu.ft.
Purity of fillmass = 94.0. Super-saturation = 1.35 @ 74.5° C and 7″ Hg abs.

$$\text{Super-saturation} = 1.35 = \frac{\text{sugar in gm. per 100 gm. water}}{\text{344 gm. sugar per 100 gm. water}}$$

Sugar in liquor = 464 gm. per 100 gm. water

$$\text{dry substance} = \frac{464}{0.94} = 494 \text{ gm.}$$

Dry substance + Water = 594 gm.

RDS (Refractometer Dry Substance) $= \frac{494}{594} \times 100 = 83.2$

Density at 83.2 RDS = 89.4#/cu.ft.

White fillmass $= 437 \times 89.4 = 39{,}068$ lb., Dry substance $= 39{,}068 \times .832 = 32{,}505$ lb.

Sugar $= 32{,}505 \times 0.94 = 30{,}555$ lb.

Volume of Crystal $= 0.7\,(6.0 \times 10^{-3})^3 = 1.52 \times 10^{-7}$ in^3/crystal

Total crystal volume $= 5 \times 10^{11} \times 1.52 \times 10^{-7} = 76{,}000$ in^3 = 44.0 cu.ft.

Density of sugar = 99.15#/cu.ft. Crystal weight $= 99.15 \times 44 = 4{,}363$ lb.

Mother liquor purity $= \frac{30{,}555 - 4{,}363}{32{,}505 - 4{,}363} \times 100 = 93.0$

Substituting $P_1 = 93.0$, $P^\circ = 94.0$, super-saturation $S = 1.35$, $t = 15$ into Equation (6). $k = \frac{\ln 94 - \ln 93}{(1.35 - 1)\,15} = \frac{0.0107}{5.25} = 0.002038$

The value of k calculated above checked fairly well with the value calculated by using initial and final mother liquor purities. Therefore, k = 0.002062 is sufficiently accurate to be used for the estimation of boiling time and final high green purity.

Changing in mean dimension of crystal with time was not linear as shown on Figure 11. Figure 11 was obtained from Figures 6, 7, 8, 9, 10 and 11. Mean dimension of crystal at one hour after graining was 0.013″ which didn't agree with the observed value of 0.016″ by Ziegler of Taylor Instrument Co. His value of 0.016″ at one hour after graining was based on linear crystal growth of 0.004″ per 15 minutes.

Substituting k = 0.002062 into Equation (4),

$$\ln P = \ln P^\circ - 0.002062\,(S - 1)\,t \quad . \; . \; . \; . \; . \; . \; . \; . \; . \; . \quad (7)$$

is obtained. If ln P is plotted against time t, a straight line with a slope of $-0.002062\,(S - 1)$ is obtained as shown on Figure 12.

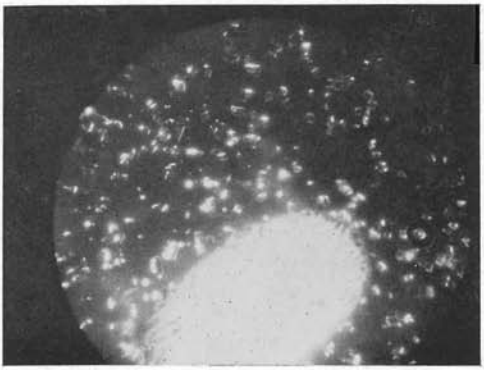

Figure 6.—15 minutes after graining. 437 cu. ft.

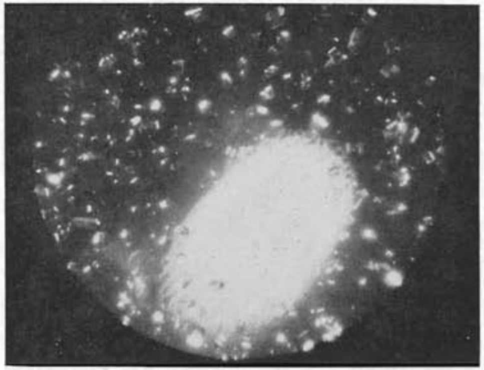

Figure 7.—20 minutes after graining. 580 cu. ft.

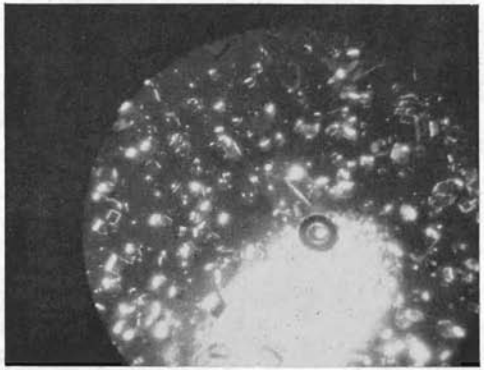

Figure 8.—25 minutes after graining. 700 cu. ft.

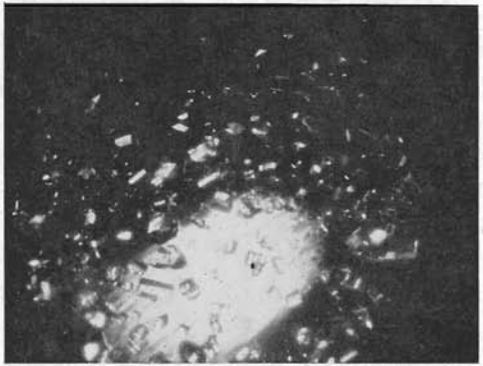

Figure 9.—40 minutes after graining. 900 cu. ft.

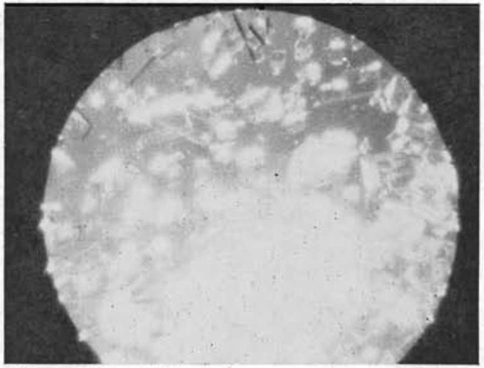

Figure 10.—110 minutes after graining. 1650 cu. ft.

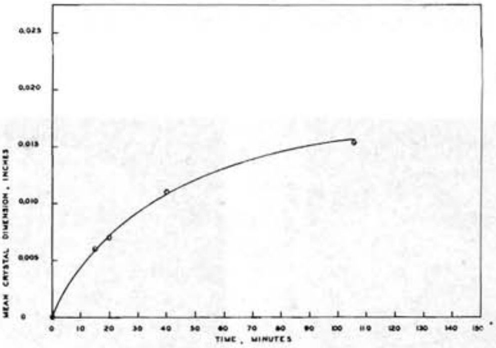

Figure 11.—Mean crystal dimension vs. boiling time.

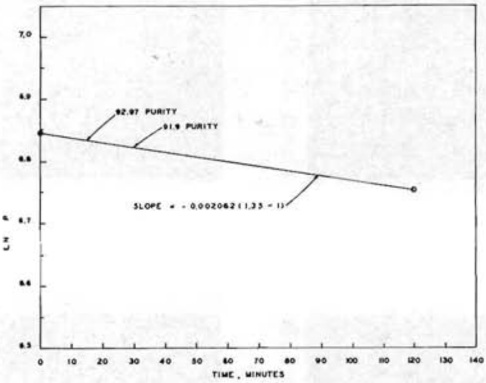

Figure 12.—LN P vs. time based on equation 7.

Final high green purity can be estimated by knowing the amount of milled fondant used and the screen analysis of sugar produced. For the experimental boiling, 200 c.c. of milled fondant was used to grain the pan and 0.016 MA sugar was obtained.

Volume of 0.016 MA crystal = $0.7\ a^3 = 0.7(1.6 \times 10^{-2})^3 = 2.87 \times 10^{-6}$ in^3/crystal

Number of crystal in 200 c.c. milled fondant = $200 \times 2.5 \times 10^9$ crystals

Total crystal volume = $5 \times 10^{11} \times 2.87 \times 10^{-6} = 1{,}435{,}000$ in^3 = 830.44 cu.ft.

Density of sugar = 99.15#/cu.ft.

Weight of crystallized sugar = $830.44 \times 99.15 = 82{,}338$ lb.

Strike volume = 1750 cu.ft. @ 91.4 RDS, 94.04#/cu.ft., 94.0 purity

Refractometer Dry Substance = $1750 \times 94.04 \times 0.914 =$ 150,417 lb.

Sugar in fillmass = $150{,}417 \times 0.94 = 141{,}392$ lb.

$$\text{High green purity} = \frac{141{,}392 - 82{,}338}{150{,}417 - 82{,}338} \times 100 = 86.7$$

$$\text{Crystallization} = \frac{82{,}338}{141{,}392} \times 100 = 59.2\%$$

High green purity calculated above checked well with the actual value of 86.2 which was determined in the laboratory. Sucrose crystallization calculated above also checked with 60% crystallization obtained in the laboratory.

Amount of milled fondant required to obtain specified MA and high green purity was shown on the following example:

Basis: 1000 cu.ft. of fillmass @ 94.0 purity, 91.4 RDS, 94.04#/cu.ft. to obtain 0.017 MA sugar and 86.0 high green purity. The pan to be boiled at constant 1.4 super-saturation.

$$\text{From Equation (7),} \quad t = \frac{\ln P^\circ - \ln P}{0.002062\,(S\text{-}1)} = \frac{\ln 94 - \ln 86}{0.002062\,(1.4\text{-}1)}$$

$$= 108 \text{ minutes} \qquad 1 \text{ hour } 48 \text{ minutes}$$

RDS in fillmass = $1000 \times 94.04 \times .914 = 85{,}953$ lb.

Sugar in fillmass = $85{,}953 \times 0.94 = 80{,}795$ lb.

Let x = lb. of sugar to be crystallized

$$86.0 = \frac{80{,}795 - x}{85{,}953 - x}\,(100)$$

Solve for x, x = 49,107 lb. of sugar to be crystallized.

Density of sugar = 99.15#/cu.ft.

$$\text{Volume of sugar to be crystallized} = \frac{49{,}107}{99.15} = 495.28 \text{ cu.ft.} =$$

855,844 cu.in.

Volume of 0.017 MA sugar = $0.7\,a^3 = 0.7\,(1.7 \times 10^{-2})^3 = 3.439 \times 10^{-6}$ cu. in. per crystal

$$\text{Number of crystal per cu.in.} = \frac{1}{3.439 \times 10^{-6}} = 290{,}782$$

Total number of crystal in fillmass to be crystallized = $855{,}844 \times 290{,}782 = 2{,}489 \times 10^{11}$

Milled fondant contains 2.5×10^{9} crystals per c.c.

$$\text{Amount of milled fondant required} = \frac{2.489 \times 10^{11}}{2.5 \times 10^{9}} = 99.6 \text{ c.c.}$$

Therefore, 99.6 c.c. of milled fondant for 1000 ft^3 strike is required to seed the pan in order to obtain 0.017 MA sugar and 86.0 high green purity for total boiling time of 1 hour 48 minutes at 1.4 constant super-saturation.

Summary

Vacuum pan boiling time can be accurately estimated by proper application of automatic controls and Equation (7) provided that the boiling is to be conducted at constant super-saturation and constant pressure.

For the given initial standard liquor and final green purities, boiling time can be greatly reduced by boiling the pan at higher super-saturation provided that the super-saturation does not exceed 1.5 which is the upper limit of Metastable zone. If the pan is to be boiled above 1.5 super-saturation, much of the fine grain formed during boiling will be so fine that they will not be retained on the centrifugal screen. They merely go through the screen and are remelted in the high green syrup. Thus, the high green purity for the pan boiling above 1.5 super-saturation will not be the same as the purity estimated by Equation (7). Equation (7) is applicable to the conditions where the sugar from the mother liquor is deposited on the existing sugar crystals and is retained on the centrifugal screen.

High green purity also can be estimated by using Equation (7), if initial thick juice purity, boiling time and constant super-saturation at which the pan is to be boiled are known.

Constant k may vary from factory to factory due to different constituents in the non-sugar and different steam pressure at calandria section of pan. Therefore, constant k is to be determined for each factory.

Nomenclature

P = Purity of mother liquor

P° = Initial standard liquor purity

S = Super-saturation

k = Constant to be determined from Equation (6)

t = Boiling time

a = Mean dimension of sugar crystal

Acknowledgment

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