SUGAR BOILING:

Some Facts and Some Fancies *by John G. Ziegler*

Quite a few years ago, a friend made the remark that sugar boiling and crystallization must be very easy or we wouldn't be able to do it. There was no disagreement among the listeners at the time but after observing hundreds of strikes boiled in all kinds of pans in many parts of the world and on many kinds of syrups, it is not hard to conclude that the quantity of good sugar boiled represents a pitifully small proportion of the total amount produced.

Sugar, to be salable, need only be sweet and have acceptable characteristics such as color and purity. Well boiled sugar sells for the same price as poorly boiled sugar. The rewards for good pan floor work lie in the lowered cost of production. Heat economy becomes of increasing importance and every ton of sugar, needlessly reboiled, represents wasted heat. Not as obvious, but of greater importance economically better utilization of existing equipment can increase pan floor capacity and cut days or weeks from the time necessary to process a crop. The tangible value of each unnecessary operating day eliminated, can justify a good deal of thought on ways in which it can be accomplished.

With these dangling rewards for improved pan floor operation it might be well to review some of the steps that can be taken toward better sugar boiling. The recommendations that follow are based on a combination of critical observation, practical experience and reasoning. They are not intended to be the final word because each year brings new experiences and situations but, coming from an engineer with quite a few years experience in many production processes, they could have the possible advantage of bringing in a detached viewpoint. After all, measurement and control principles worked out for the sugar industry have been applied with rewarding results to many others, chemical, petroleum or even aerospace so why not reverse the process for the benefit of the sugar industry. It is worth a try.

A considerable advance was made some years ago with the development of pan microscopes capable of displaying crystal growth within the pan itself. That tool helped to prove and disprove many beliefs based on visual examination of proof samples withdrawn from boiling massecuites. Many of the conclusions presented here are based on thousands of observations made with pan microscopes during the course of boiling all grades of beet and cane syrups in most types of vacuum pans.

SUPERSATURATION

The most important variable in sugar boiling is the degree of syrup supersaturation so a brief review is in order. A syrup of given purity at some temperature is said to be saturated when the concentration is such that sugar crystals in contact with it neither dissolve or grow. Increasing the syrup concentration or lowering its temperature makes it supersaturated and suspended crystals will grow. The degree of supersaturation is usually expressed as the weight of dissolved solids per unit of water, divided by the weight that would be in a saturated solution at the same temperature. The ratio is 1.0 at saturation. Since values below saturation are of little interest in sugar boiling where the object is to grow sugar crystals, a handier unit to use is the degree of "Oversaturation"; a solution at 1.2 supersaturation is 20% oversaturated or 20% O.S. in the interest of brevity.

Crystal growth rate appears to be almost directly proportional to the oversaturation of the surrounding syrup according to many investigators. But there is an upper limit of oversaturation beyond which the driving force is too great for orderly growth of existing crystals; tiny new ones form spontaneously in the surrounding syrup. This so-called "false grain" formation must be avoided in all stages of the boiling process if good grain and high yields are to be obtained.

The useful syrup oversaturation range called the "metastable" zone, lies between saturation and the concentration at which false grain forms; any higher concentration takes it into the "labile" zone where new grain is inevitably produced. Using the latest saturation and boiling point data coupled with many observations, the boundary between the two zones seems to fall right around 65% O.S.

FACTS OR FANCIES

In the copious literature on sugar boiling, one finds several recurring ideas expressed which do not stand up entirely in the light of careful investigation. It would be well to examine them individually if conclusions are to be reached about optimum sugar boiling techniques.

INTERMEDIATE OVERSATURATION ZONE?

One idea is repeatedly advanced that there is a twilight area of oversaturation lying between the metastable and labile zones; an area in which new grain will form in the presence of existing grain but not in syrup alone. It is possible that such a state might be produced in the laboratory but it has not been possible to prove its existence in any actual vacuum pan. Repeated tests on various syrups and continuous microscope observation have verified that nucleation takes place at the same concentration whether grain is present or not. The reason for the persistence of the idea is probably due to the difficulty of observing the presence of new nuclei. Various sources report that the smallest particle that can be distinguished with the unaided eye is around 40 micron in size. On a proof slide viewed against a light in the normal manner, a somewhat smaller crystal might be visible due to sparkle from crystal facets; a good estimate might be 25 microns or 0.001" size.

Microscope observations reveal that in fairly pure syrups, crystals will grow at the rate of about 0.015”/hr. on the mean dimension when the syrups are just below the 65% O.S. limit. This is equivalent to a sugar deposition rate of 375 microns/hr./sq. meter and agrees well with the highest growth rate reported by others. (1). At this rate, it would take 4 minutes for a newly formed particle to become visible to the naked eye but only 5 seconds under a 50 power microscope. Further verification comes from repeated experiences of sugar boilers exceeding safe O.S. values; new nuclei are visible almost instantly in a pan microscope but, the boiler will not agree for 4 or 5 minutes that there are indeed new grain visible on his proof slide.
CONCLUSION: No intermediate O.S. zone exists.

DOES AIR LEAKING IN TO A PAN FORM ADDITIONAL GRAIN?

Instructions for seeding pans often caution against allowing any air leakage which reputedly will form additional grain. The fallacy of this is easily proved if a charge of syrup is held just below the labile zone by feeding water and a copious quantity of air admitted by opening a seeding valve or pulling out the proof stick. Air has been bubbled for minutes through such concentrated syrup without a single grain being observed in pan microscopes. Neither will live steam now through the syrup form grain. CONCLUSION: Only excessive oversaturation forms new grain.

DO MECHANICAL CIRCULATORS FORM GRAIN?

It is occasionally reported that successive strikes boiled in a pan, using its mechanical circulator on one strike and not on the other, will invariably produce finer grain on the circulated one; this in spite of careful boiling and identical quantities of seed. The easy conclusion is that the circulator formed additional nuclei. Examination of several such cases reveal an entirely different reason. The higher degree of agitation induced by the circulator in the early stages of the strike simply reduced the number of seed crystals that stuck together to form conglomerate grain. More of the individual seed survived so the average size in the finished massecuite was smaller.
CONCLUSION: No evidence that they do.

DO HIGHER BOILING TEMPERATURES PRODUCE HARDER GRAIN?

The sugar boiler's finger can sometimes detect a difference in sugar quality; he will say that it is pretty soft grain or "good hard grain". A pan microscope reveals that his hard grain is only clean grain as opposed to balled or conglomerate crystals. Equally clean grain strikes can be boiled at any temperature.

There has to be a reason for the belief that higher temperatures are to be preferred. The metastable zone of oversaturation covers a considerably wider range of concentration at higher boiling pressures so it is proposed that in his experience, the sugar boiler found that he was able to boil more strikes of "hard" grain at high pressure than at low .
CONCLUSION: Not true.

IS CRYSTAL GROWTH FASTER AT HIGHER TEMPERATURES?

Test results by Kamoda and Yamane (2) and others indicate that the growth rate of sugar crystals is increased somewhat at elevated temperatures. Test results in this study do not prove or disprove those observations and no apologies are offered for this reason: On any one pan and syrup, equipment and facility constraints do not permit boiling a production pan over a very wide range of absolute pressure. On the low side, one is limited by available water temperature and condenser capacity and vaporization rate from the pan must be penalized. On the high side, the available temperature difference between steam and massecuite or considerations of color formation limit the experimenter. In general, the allowable variation in boiling temperature is quite limited in any one situation.

Perhaps the question can be answered by reducing it to absurdity. Most diffusion processes proceed more rapidly as temperatures are increased, simply because fluidity normally increases with temperature. If the predominant mechanism governing the rate of sugar molecule migration to growing crystal faces is syrup fluidity, which seems likely, it must be pointed out that the fluidity of a given syrup boiling at 10" Hg. Abs. near the upper limit of the metastable zone has a fluidity only 60% higher than it would have under the same condition at 4" Hg. Abs. These pressures represent almost the outside limits used in practical sugar boiling so it must be concluded that the tolerable variations in any one situation can have only a negligible effect on crystallization rate.
CONCLUSION: There are far more important considerations.

DOES WATER FEED TO A PAN WASH OUT GRAIN?

This conjecture has been voiced quite often because it is a logical assumption, that the smaller crystals in a massecuite would be quickly annihilated by contact with a stream of water fed to a pan to maintain syrup condition during intervals of inadequate feed syrup supply or when it became necessary to provide time for existing crystals to grow.

If, at any time, syrup in a pan falls appreciably below saturation, grain will start to dissolve and this is immediately apparent in a pan microscope because crystals with rounded corners are observed. But microscope observations of pans boiling on water for extended periods have never revealed a single grain showing such damage. Apparently if the bulk of the pan syrup remains somewhat oversaturated, the entering water simply cannot penetrate the viscous film of syrup surrounding each crystal before it mixes with the more concentrated pan liquor.
CONCLUSION: Probably untrue.

WILL A DRINK OF WATER OR SYRUP WASH OUT FALSE GRAIN?

When a proof slide reveals the presence of false grain, most sugar boilers will increase feed rate temporarily or add a "drink" of water feed for a minute or so. A few minutes later a sample appears to indicate that the mishap has cleared up. The "drink" may well drop the O.S. back into the safe metastable zone but, unless it is diluted to a concentration below saturation, no grain will be dissolved. The pan microscope reveals what really happens. The new grain, in a few minutes, grow to the size where conglomeration occurs. They form ball grain and approach the size of the regular crystal crop. To the sugar boiler's unaided eye, it appears that he has washed out the fine grain but in reality he has fouled the nest and the strike will consist of a large percentage of inferior sugar.

It would require a copious drink of water to drop 65% O.S. syrup below zero. For example, in a beet white strike at 93 purity boiling at 7" Hg. Abs., suppose the O.S. limit is exceeded when syrup volume is 700 cubic feet. Approximately 87 C.F. of water would have to be added to reduce pan contents to saturation plus the amount that had evaporated during the time necessary to introduce it. Plus the amount that would be required to dissolve the new crystals. The quantity of 70 brix liquor required would be around 250 C.F.
About the only way to eliminate false grain once formed in an established strike is to add sufficient water to drop syrup well below the O.S. limit, break vacuum and heat up the massecuite until it is well below saturation. When the newly formed grain have disappeared and the established crystals all show rounded corners, the pan can be returned to normal operating pressure and boiling continued. But even if this is done well, many minutes of useful work are lost.
CONCLUSION: False grain is not eliminated by a drink of water or syrup.

DOES A TIGHT MASSECUITE PRODUCE BETTER GRAIN?

Another recurring notion is that a "tight" massecuite (high crystal to liquor ratio) makes better grain than a "loose" one because adjacent crystals, in rubbing together, will improve their form and growth. This is not confirmed by experiment. If anything, strikes boiled on the loose side show better grain formation under the microscope, possibly because lower massecuite consistencies enhance overall pan circulation, promoting less size variation in final crystals.
The belief probably came about because sugar boilers, over the years, discovered that the odds of producing better sugar were increased if they carried the massecuite on the heavy side. It's logical, because the resultant lower heat transfer minimized the chances of exceeding safe O.S. concentrations. But experiments conducted on successive strikes, while not conclusive, indicate that better grain formation and crystal uniformity are obtained if crystal yields are held in the low (15% to 20%) range during the growing period up until final concentration to dropping consistency.
CONCLUSION: An unconfirmed conclusion.

SHOULD MASSECUITES BE GRADUALLY TIGHTENED AS LEVEL INCREASES?

An argument on this subject is as futile as the classic one regarding how many fairies could dance on the head of a pin. If agreement can be reached on the area of dance floor required for each fairy to dance and on the area of an average pinhead, there can be no disagreement between engineers capable of wielding a slide rule.

The time required to boil a strike from graining to dropping is determined by either the time necessary to evaporate the water or the time needed to grow crystals of the desired size. With low heat transfer or thin syrup, time is saved by carrying lower consistencies to obtain high heat transfer and better circulation. On the other hand when heat transfer is good, feed syrup is heavy and large grain are wanted, it is desirable to hold high O. S. to hasten grain growth and allow massecuite consistency to increase as the pan fills, penalizing circulation.
CONCLUSION: It depends on operating conditions.

IS "SHOCK" SEEDING A REALITY?

This is a controversial one. Where full fondant seeding techniques are not practiced, the so-called "shock" method is used to initiate grain. A syrup charge is concentrated and a small charge of powdered sugar is introduced; boiling is continued until a proof slide appears to have sufficient grain, whereupon syrup feed is started, usually with a "drink" to stop further grain formation. Supposedly, the sugar by its mere presence "shocked" the unstable syrup into spontaneous nucleation to form most of the needed grain.

But as previously noted, there seems to be no intermediate oversaturation zone in which nucleation will take place only in the presence of existing crystals. So whence came the notion of shock seeding? It was a reasonable conclusion based on tests and observation without proper visual aids but microscope monitoring of many strikes supposedly shock seeded made it apparent that no such thing happened. Generally the sugar boiler carried the syrup in to the labile zone grain was already forming but too small to be seen. In a short time he could see the larger particles of the shocking grain and it comforted him by proving that he had correctly judged that the syrup was adequately concentrated or they would have dissolved. Usually by continuing to boil he went farther into the labile zone and far too many nuclei were formed and easily visible in the pan microscope. As they increased in size the remarkable tendency of sugar massecuites to heal themselves stepped in and they conglomerated forming larger clumps of grain. When this occurred, the sugar boiler could begin to see them on his slide and when he judged the crop was adequate, gave a drink of syrup which brought him back into the metastable zone.

Other strikes were observed where the "shocking" sugar was introduced earlier with the syrup in the metastable zone. But boiling soon concentrated it to the point that nucleation took place as before and all the sugar boiler noticed was that it took another minute or two for the "grain to come in".

The great shock seeding myth was further perpetuated by the reports of accredited experimenters who ran careful elutriation tests on dry fine sugar samples of the material being used to initiate grain. The result was that it would take an inordinate amount of the fondant to full seed a pan because the analysis showed that the amount being used contained only about 10% of the required nuclei. Q.E.D., the remaining 90% had to be produced by shocking unstable syrups.

Careful experiments indicated that there was something wrong. Pans were seeded up toward the limit of oversaturation with a weighed amount of hammer-milled fondant sugar and grown to maturity being careful to avoid further nucleation. The mean aperture of final crystals was determined. Succeeding strikes of the same syrup were seeded with the same amount of fondant but held well below the oversaturation limit until the danger of further nucleation was past. Surprisingly, the mean apertures were essentially identical to those of the strikes supposedly shocked at higher oversaturations; the crystals should have been over twice as large if in reality there had been only 10% as many. The only possible conclusion was that all the required grain had been introduced in the fondant.

For this experiment, the hammermilled fondant had been independently analyzed by a University laboratory. They reported so many 0-5 micron particles, so many 5-10 micron, etc. per gram and it was no problem to calculate the total number of nuclei per gram. But apparently only the magic 10% were present in the amount of fondant being used. Where did the additional 90% come from?

A slide rule exercise indicated a most logical explanation for the discrepancy. Any sugar particle is a viable nucleus for growth of a crystal in an oversaturated sugar solution. Suppose that some dust particles averaging 0.2 micron in size had been present in the fondant and in the elutriation test had been blown away by stray laboratory drafts or had been overlooked and not counted on the first test slides. How many of these tiny particles would it take to provide the missing 90% of pan grain?

In a 1500 C.F. strike of sugar with mean aperture of 0.015" (0.4 mm) approximately 3.5 x 10^{11} grain would be produced if final yield were a reasonable 55% of total massecuite solids. Assuming crystals were cubical to simplify calculation, the amount of 0.2 micron seed required to supply the missing 90% turns out to be a mere 4.4 milligrams. This bare pinch could easily be present in a kilogram or so of dry fondant.
CONCLUSION: There is no such thing as "Shock Seeding".

THE IMPOSSIBLE DREAM

In sugar boiling, the ultimate goal is to boil each strike in minimum time and produce the highest possible yield of crystals, all of uniform and the desired size, and each crystal perfectly formed, completely free of balled and conglomerate grain. Such a massecuite would be very fluid even at high yield, would purge quickly in centrifugal and require a minimum amount of washing. The sugar would dry readily and store well.

Unhappily, even the best operation of conventional vacuum pans cannot produce such perfect grain. Some pans are better than others but all fall far short of perfection. The purpose here is to point out the factors involved in operation of pans, imperfect though they may be, that can improve the quality of work done in them and approach ideal operation.

UNIFORM GRAIN SIZE

One of the first objectives in sugar boiling is to produce grain with the least possible variation in size. Uniform crystals purge readily in centrifugals because passages thru the bed are large in area. But if smaller crystals are present, they fill the interstices and retard the flow of syrup. The wider the variation of crystal size, the poorer the purging characteristics.

A good measure of grain uniformity is called the "Coefficient of Variability" which, though arbitrary, puts numbers on the variable and makes possible comparison of sugar quality with regard to size variation. It is defined as 100 times the difference between screen aperatures that would retain 16% and 84% of a sample, divided by two times the "mean aperture," which is the size that would retain 50% of the sample. The two measures are abbreviated Cv and M.A. Good operation of a good pan will produce sugar, as boiled, with Cv in the low 20's which is excellent. Even poor pans, if operated well, can hold Cv below 30 which is not bad. A Cv of 40 represents inferior work and higher values indicate very poor sugar indeed.

PAN CIRCULATION

If all crystals in a pan were subjected to the same syrup environment during the course of a strike, they would grow at very nearly the same rate and reach about the same final size. But the circulation pattern in vacuum pans is such that oversaturation gradients exist and crystals that spend more time in lower O.S. syrups do not grow as fast as those in areas of greater driving force. The circulation pattern of a pan limits the minimum Cv that can be obtained.

The conventional picture of circulation in a calandria vacuum pan has the heated massecuite rising in orderly manner to the surface, across and down thru the center well for reheating. Critical observation discloses that ebullition is more often near the center of the pan and massecuite flows outward at the surface. This stream flows downward and, meeting material rising from the tubes, forces it in toward the center where it may flow back down the center well without ever reaching the top surface to flash down to the pressure at that point. Thus, the cross section of flow is more of a figure eight. The lowest temperature will be at the surface where the pressure is-lowest; the material that by-passes to the center well will be warmer and consequently at a lower oversaturation.

Quite often one observes a temperature just above the calandria, 2 to 4°C higher than at the surface and this represents a considerable drop in oversaturation since the entire metastable range of a syrup from saturation to the upper limit only requires a change of 4° to 6°C depending on purity.

Mechanical circulators improve the flow pattern somewhat but do not cure it since forced circulation is only effective for a few inches above the top of the calandria. The percolating effect of boiling is the only real mixing force above that. Due to the low velocities involved, the addition of louvers to a pan seem to have no effect whatever in changing circulation patterns or improving Cv.

Of the many pans included in this study, straight sided ones consistently performed better than so-called "low head" pans with shells larger than the calandria diameter. This has been confirmed by Rogers (2).

Circulation patterns can be improved in a calandria pan if feed syrups are maintained at elevated temperatures and introduced through suitable distributors to take advantage of the resulting flash of vapor. Introduction at several points around the pan under the calandria and at or near the outer periphery, induces more "outside-in" circulation than would other wise take place.

Circulation can sometimes be bettered by boiling at lower absolute pressure to increase the volume of vapor but the limit is reached when boiling rate must be reduced to stay within operating bounds fixed by the condenser water temperature and the ability of a condenser to handle high water flows.

No comments can be made at this time regarding the circulation pattern of pans equipped with floating calandrias or of horizontal pans for the simple reason that they are not often encountered and there has been no opportunity to study them.

FIGHTING THE ODDS

From the foregoing it should be apparent that it just isn't possible to boil a perfect strike of sugar in any vacuum pan in use now or one likely to be devised in the foreseeable future. But there are still techniques that can be applied to existing equipment which can approach the impossible dream and vastly improve present technology of sugar boiling. Once the more desirable procedures are recognized and put into effect, immediate benefits are realized; and they need not be expensive modifications of present poor operating methods.

This present exposition must necessarily be limited in scope and will be directed primarily at the areas of greatest possible return which include the boiling of higher purity finished sugars in refineries, beet sugar factories and cane mills. The principles apply equally well to the boiling of lower purity massecuites but in those areas, the problems of pan floor management can often confuse the basic pattern. Later, perhaps, a discussion of the low purity end of sugar boiling would be in order.

CONGLOMERATION

In low grade syrups, there seems to be little tendency for crystals to stick together and grow as one forming what is known as conglomerate grain. But in higher purity syrups, it becomes a definite problem. A conglomerate crystal is of lower quality than a single clean crystal for several reasons which will be pointed out, so it is highly desirable that everything possible be done to minimize the number of unclean crystals produced.

Increased conglomeration in a properly seeded strike, reduces the number of final crystals, or rather the number of particles produced so the average size, M.A., increases. Likewise the Cv increases because the conglomerate grain gets larger in size at the expense of individual clean crystals. Thus increased conglomeration detracts from the number of properly sized grain and, in increasing the Cv, detracts from the purging qualities of a massecuite .

There is not very conclusive evidence that impurities are included between faces of crystals making up conglomerate grain but they are inferior to clean grain in their purging, washing and drying characteristics. A single crystal has large angles between faces but a conglomerate will often have smaller angles where grains join, and in these corners, a fillet of syrup is held by surface tension. More washing is required to attain proper color and purity of finished sugar which, of course, means more wash syrups to be reboiled. Also, after washing, fillets of wash syrup adhering to conglomerate crystals increase drying problems; a skin of dried syrup forms and traps a bit of moisture which will eventually diffuse out thru the dried fillet. In bulk storage this released moisture will migrate to cooler areas and can cause serious caking.

Kamoda and Yamane (3) made a meticulous study of the factors affecting conglomeration and their results are amply confirmed by microscope observation of actual pan massecuites. The combined conclusions are briefly enumerated here.
1) Conglomerate grain formation is directly proportional to the degree of oversaturation of the surrounding syrup.
2) Conglomerate formation is inversely proportional to the mean space between growing crystals.
3) Conglomeration is inversely related to the degree of agitation of the syrup and crystals; not a straight line but it is certainly reduced by increasing agitation.
4) Conglomeration increases with syrup purity.
5) Conglomeration is affected by crystal size; it seems to occur over a relatively narrow range around 0.001" (25 micron) and drops off abruptly on either side of this.

Mechanically, there is no reason to disagree with the observed conglomeration factors. Certainly, two crystal facets which came together would have more tendency to fuse in a highly oversaturated, high purity syrup if they were not separated by syrup movement before the union took place. In a sparse crystal crop, the chances of two crystals meeting in proper conjunction for conglomeration would be poor.

The narrow size range in which conglomeration takes place would not have been as easy to predict but it is not illogical. The chance of collision would certainly be greater as crystal size increases. The reason that it drops off with further increase in size is probably due to a combination of factors; the increasing area presented to agitation forces could well separate crystals before growth welded them together. Then too, small crystals have beautifully flat facets but irregular growth patterns make them rougher as they increase in size. So the chances of faces wringing together should decrease with size.

Conglomeration should be minimized but reexamination of the factors affecting it reveal a limited number of possible courses to pursue. Little can be done to increase the mean distance between crystals in a graining charge except to increase the volume and this is self-defeating because it leaves less volume for feeding during the growing period and reduces the time available for grain growth. Doubling the graining charge only reduces the mean distance by the cube root or 26% and this is negligible.

The purity angle has some possibility. A few cubic feet of low purity, highly oversaturated syrup could be fondant seeded and cured with excellent agitation for a reasonable time so that the seed would grow larger than the size for conglomeration. This slurry, introduced as seed into even a high purity strike would have a negligible effect on the overall pan purity but would reduce conglomeration to the vanishing point. However, the manipulation required would undoubtedly become too complex for day to day processing.

So, practically, about the only variables that can be manipulated readily are agitation and oversaturation. In the interest of reducing conglomeration it is thus advisable to maintain maximum agitation (mechanical circulation plus maximum boiling rate) until grain has passed the critical conglomerating stage, but this is more or less fixed by facilities and equipment. That leaves only the variable of oversaturation that can easily be manipulated. A sugar boiler with only his crude estimation of oversaturation provided by the feel and look of syrup viscosity at graining time, tends to hold high values to preclude melting out the newly formed grain. But with a good measurement of the variable, it is no problem to seed and hold oversaturation in the 40 to 50% range for a few minutes until the conglomerating stage is passed and produce a basis of clean individual crystals. With good operation at this stage of the cycle, it is possible to essentially eliminate conglomerate grain in the final sugar. In the total strike time, a very few minutes spent in establishing a footing of good individual grain are well worth while because then, oversaturation can be increased toward the maximum for fast growth and resulting sugar quality vastly improved.

SEEDING

Grain formation can be initiated in various ways. The shock seeding method has already been exploded; it only takes an excursion into the labile zone of oversaturation to form nuclei. Experiments have been run in an effort to produce a reproducible crop of initial crystals by precise regulation of the time and extent of the excursion into the labile zone. The results have been hopelessly disappointing. The only successful method as of now seems to be that of seeding an oversaturated syrup with a measured amount of standardized very finely ground sugar slurry.

Ordinary powdered sugar from screenings is not too satisfactory as it can vary from day to day and excessive quantities may be required. Dry fondant sugar prepared by milling reduces the quantity but the size distribution is difficult to reproduce and it does not store well; particles stick together due to temperature and humidity changes. Even vigorous shaking into a slurry with isopropyl alcohol will not separate adhering crystals and the result is that many already conglomerated grain are introduced to the pan in the seed itself. It has been reported that such slurries are improved in this respect if subjected to vigorous mixing in a high speed blender but no quantitative information is available .

At this time it appears that the best seed slurry is obtainable by wet grinding sugar in some medium like isopropyl alcohol. Ordinary laboratory ball mills are widely used. A mixture alcohol and sugar in approximately 2/1 volumetric proportions, if ground for 12 hours or more approach an ultimate fineness and contain about 2.5×10^9 particles per ml. More sophisticated devices such as the "Ditmar" or "Sweco" mills will achieve the same result in less time. In any event the slurries are stable for indefinite periods and if the grinding procedure is standardized, vary little from batch to batch.

Other vehicles such as linseed and cottonseed oil have been used. Commercial Ethyl alcohol was used in the tropics because it was readily available from adjacent rum distilleries but reportedly was less stable with time because of the 5% water in the alcohol; temperature changes would cause the finer particles to dissolve and accrete on the larger ones. Anhydrous isopropyl alcohol which is inexpensive and almost universally available is an ideal medium.

The quantity of seed slurry to use can be predicted with fair accuracy and corrected on succeeding strikes to obtain the desired M.A. As a guide, a 1500 C.F. pan of white sugar, if well boiled, can easily give a 55% yield of crystals from total solids. 150 to 200 ml. of ball milled slurry will be adequate for 0.015" M.A. grain.

The quantity needed varies directly with massecuite volume and inversely as the cube of crystal size. Small changes in seed quantity have a negligible effect on mean aperture of finished sugar. In theory at least, eight times as much seed slurry would be required to halve the M.A.

It is even less precise than that. To get smaller crystals, more than the predicted amount of seed must be used because, with the decreased mean space between crystals, additional inevitable conglomeration does deplete the crop.

An even more important variable in seeding must be recognized and that is the oversaturation prevailing at the time of seeding and shortly thereafter. It has already been pointed out that syrup oversaturation can vary considerably from point to point in a vacuum pan. In a graining charges boiling vigorously, the bulk of the liquor can be very uniform but there will exist limited areas where the climate is one of dissolution rather than growth, specifically adjacent to heating tube walls. Larger seed particles can stand an excursion of limited duration thru such zones but not the tiny ones. In order to duplicate the number of surviving nuclei from strike to strike, the oversaturation level at the time of seeding and for the following few minutes is of greater importance than the amount of seed introduced. This has been verified in practice. But it is quite surprising that by using reasonably standardized seed and following the same oversaturation pattern that strike after strike can be dropped varying less than 0.001" in mean aperture.

FALSE GRAIN

Once a strike is seeded and a footing of grain free of conglomerates is established, it is safe on first base. But lack of care at any time thereafter can cause damage that cannot be easily repaired without excessive loss in production and pan floor economy. False grain formation at any stage of boiling detracts from the quality of final sugar. True, at certain stages it causes less economic damage than at others, but it must never happen if the rewards of good pan operation are to be had .

When first seeded, the crystal area available for sugar deposition is around 200 sq. ft. and, at best, only a fraction of a pound of sugar per minute is being added. During final concentration, the area amounts to several million square feet and the sugar yield in a normal strike can be well over a ton per minute. In the early stages practically all the water being evaporated must be supplied by that coming in with the feed to raise it to the concentration required for the necessary oversaturation. As crystal area increases, more of the evaporation is allocated to liberating sugar for crystallization. Hopefully, during the final concentrating step, enough crystal area will be available to absorb all the sugar being liberated by evaporation of water from only the syrup in the massecuite .

False grain formed in the early stages of a strike simply conglomerates to form fragile ball grain as has been discussed. Unless melted out at the expense of time and steam, it continues to grow along with the true grain, affecting the M.A. and lowering the quality of the final sugar in several ways. Formed farther along in a strike, new grain will lower M.A. and increase the Cv to cause even greater reduction in sugar quality and yield.

There is real danger that false grain produced in the middle stages of a strike may not have time to grow sufficiently and may only attain the size that effectively plugs centrifugal screen openings so that a massecuite will not purge and the entire strike must be remelted.

In a typical strike, carried in text book fashion through the seeding and early growing stages until maximum oversaturation can be employed to hasten grain growth, will begin to increase in consistency. It is often called "coming together". The crystal/ liquor ratio increases to the point that overall viscosity is greater than that of the syrup itself due to the presence of the 15% to 20% yield of crystals. To prevent undue increase in consistency which would detract from the overall massecuite circulation and heat transfer, syrup feed is increased to maintain consistency and oversaturation of the syrup slowly falls.

When maximum massecuite level is reached and syrup feed is withdrawn, consistency begins to increase and so does oversaturation. If it goes beyond the safe limit, false grain will form. It doesn't ruin an otherwise good strike or detract too much from sugar quality but it represents a horrid economic waste .

Sugar boilers, pressed for time, often force the final concentrating step so that a massecuite can be dropped and another one started. This is unbelievable folly. False grain formed during the last few minutes of a pan cycle certainly do speed the increase to dropping consistency; the myriad small grain formed, increase syrup viscosity so much that it appears the crystal yield is rapidly increasing to the dropping point. But it is not a yield of usable crystals, only fine trash which will nearly all go through centrifugal screens and require remelting and recrystallizing. They add to syrup viscosity and slow purging of the good crystals. And those tiny grains adhere to the otherwise clean crystal surfaces increase drying difficulties and generally wind up as dust in drying and storage due to attrition.

So much better to stay below safe oversaturation concentrations during final concentration, taking a few more minutes and putting a few more tons of sugar on existing crystals and not leaving them to be reboiled over and over on the pan floor. Better to maintain syrups in the safe metastable range by reducing steam flow or better adding some water feed. A little extra time spent in the final, most productive stage of sugar boiling is not wasted, but amply rewarded by the increased yields and reduction of wasteful recirculating loads.

CURING

The current fetish about possible color formation or massecuite deterioration in prolonged storage has led to decreased volumes in pan receiving vessels or mixers. This could be a distinct step backward. A cold look from the practical point of view indicates that considerably increased yields could be realized from modest retention times of massecuites prior to centrifugal processing. This is well recognized in low grade work where pan capacity is always inadequate to hold massecuites until they can approach maximum yield; crystallizers are then employed, not so much to maintain adequate oversaturation levels by gradual cooling, but to give time for crystal growth in lower purity syrups.

A typical white strike of beet or cane sugar is generally highly oversaturated when dropped to the mixer, often just short of the limit. A 40% yield strike with the syrup 50% oversaturated is still depositing sugar and will approach 60% yield as the syrup falls toward saturation. Instead of operating the centrifugal station to process a strike in the least possible time, it is better to work at a more leisurely pace leaving only enough space in the mixer to accept the following pan when it is ready to drop. This was pointed out to a Hawaiian mill some years ago where they were blessed with a 2-1/2 strike mixer. By regulating their centrifugal operation to work toward a full mixer instead of an empty one, several tons of additional sugar were recovered from each strike .

In fairly high purity syrups, the rate of sugar deposition is such that the syrup falls toward saturation on about a 15 minute time constant; in the first 15 minutes, 63% of the available sugar is recovered and 86% at the end of 30 minutes. By utilizing all possible storage capacity to prolong curing time, the sugar yield per strike can be increased by impressive amounts.

Some boilers attempt to squeeze additional sugar out of the syrup by lowering pan temperature before or during final concentrating. True, at lower temperature the syrup will be less concentrated but it is a very dangerous procedure without a very reliable oversaturation measurement. In the final stage of a strike, the oversaturation is generally crowding the upper limit and any drop in temperature will raise it into the labile zone; more sugar will be lost as false grain than could be gained by lowering syrup concentration. Even if the lowering of boiling pressure is done safely, the gain in sugar yield is small compared to that available by extending curing time.

OVERSATURATION MEASUREMENT

From the foregoing, it should be apparent that oversaturation is the most important variable in sugar boiling. If held at a favorable level in the early stage of grain growth, a base of clean individual crystals can be produced which is the first step in the production of high yield strikes of quality sugar. After danger of conglomeration is past, high rates of sugar deposition are obtained by holding syrups just short of the labile zone and final concentration to dropping brix without wasteful formation of fine grain can be accomplished in minimum time if the oversaturation is held high but safe.

Absolute pressure or vacuum is of lesser importance as long as it is held within reasonable limits and not allowed to very abruptly, but really only so that oversaturation can be maintained at the most desirable point at all times. Consistency is important to a degree in the middle portion of a strike after grain has "come together" but considerable latitude in this variable has little effect on economy and quality. Massecuite level at the low or high end can stand some variation without materially affecting operating efficiency. Steam flow need only be kept within the bounds of condenser capacity or to keep from outraging the capabilities of the boiler house or the operation of evaporators when vapors are used.

A sugar boiler judges the degree of syrup oversaturation by various means but they are all based on the viscosity of the syrup which does vary over a fair range between saturation and the limit between metastable and labile zones. From microscope monitoring of strikes boiled by even the most experienced sugar boilers and double checking with measuring instruments, it appears doubtful they are able to judge the true oversaturation of a familiar syrup within plus or minus 25% and this is not close enough to do better than very mediocre boiling. His "feel" deteriorates once crystals are present because they affect the overall massecuite viscosity making it higher than that of the syrup alone.

Several measuring devices and methods have been used for many years to supplement the sugar boilers judgment of syrup oversaturation. One, still widely used, is the electrical conductivity of boiling syrups and massecuites. This property is a useful rough guide but is not specific since its reading changes considerably with crystal crop, soluble electrolyte content of the syrup, etc. (1). Refractive index of boiling syrups has been used to measure their concentration but, according to reports, presents mechanical and maintenance difficulties and is a slow measurement, due to the time required to change the viscous syrup film adjacent to measuring prisms.

For many years schemes based on variations in boiling temperature have been used with some success. At a given pressure, the boiling point of a syrup changes predictably with its concentration and can be used to monitor oversaturation with adequate accuracy if properly applied. The temperature change over the metastable zone is only 4 to 6^{O}C but this poses little problem nowadays with currently available pneumatic or electronic transmitters which can cover quite narrow temperature spans. Equally good readability of absolute pressure is required if the oversaturation is to be determined from the combination of the two variables.

The boiling pressure-temperature method is not universally adaptable and, as often applied, has serious limitations and pitfalls. In the first place, absolute pressure must be controlled very precisely and this is not easy to do unless barometric condensers are of good design and capable of close regulation; and many are not. Plants equipped with central condenser systems are almost impossible to regulate within the required limits because intolerable pressure variations occur each time a pan is connected or disconnected from the central condenser. Individual "water jet" condensers often do not have sufficient turndown ratio to handle the varying vapor load throughout a strike, making it impossible to maintain precise pressure. Compensation of the temperature reading for changes in pressure is very complex due to the considerable curvature of the vapor pressure curve over the normal boiling pressure range plus the changing slope of oversaturation values at various pressures.

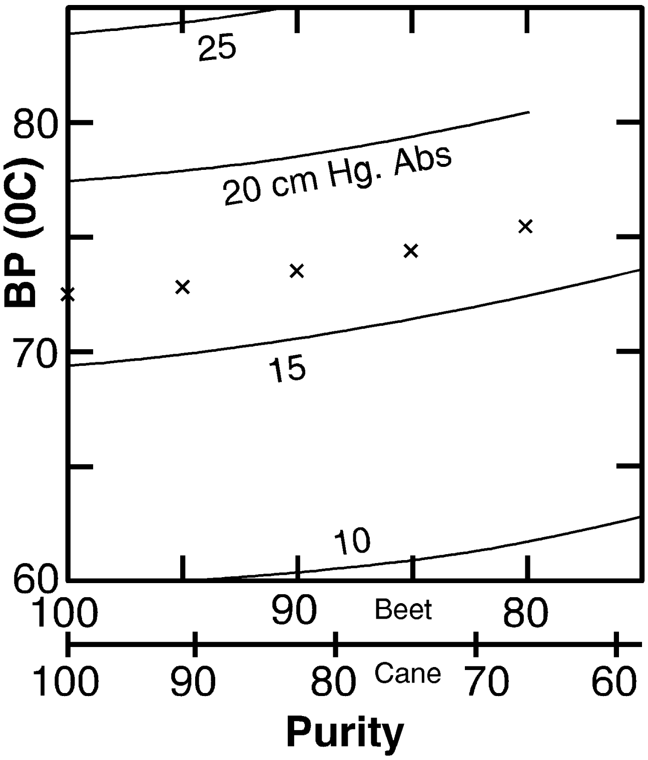
The greatest difficulty and limitation associated with boiling point measurements for many years was the problem of measuring the temperature at the point of highest oversaturation. Conventionally the bulb has been located in the upper part of calandria pan center wells so it would be immersed even during concentration of the low graining charge. This was an excellent location during the early part of the strike but, as level increased, by-passing of massecuite made the readings increasingly arbitrary depending on circulation vagaries so it quickly became meaningless and useless for the remainder of the strike.

As previously noted, the lowest temperature and so highest oversaturation in a vacuum pan must exist at the boiling surface where the lowest pressure exists. The problem of mechanically keeping a temperature measuring bulb at the surface or shifting to multiple bulbs seems excessively complex. But vapor leaving the upper surface is superheated and at the temperature of the massecuite at that point so it was found that a sensitive bulb, located at a point of good velocity in the pan entrainment separator or vapor line gave a reliable measurement of the minimum boiling temperature in spite of varying level. Much use has been made of this method during the past years and it has contributed significantly to the quality of pan work in many plants.

Pending some break-thru on a measuring element that really measures the "priceless ingredient" of oversaturation or the degree of tension toward formation of spontaneous grain, it appears that the best solution at this time lies in temperature measurement of vapor at a known boiling pressure. Where absolute pressure can be precisely controlled, a sugar boiler can be easily trained to always stay below one definite temperature to avoid false grain formation; this has been done even in some "developing" countries. But where equipment and facility frail ties make tight pressure control unreal, it becomes most difficult to provide a chart or table with which he can quickly correlate the temperature limit in terms of changing pressure. And do it with sufficient speed and accuracy to prevent errors.

The boiling points of typical beet and cane syrups at 65% oversaturation are plotted in Figure 1 for several absolute pressure values. Although the temperature change with purity appears to be small, it is quite appreciable compared to the available temperature range over the metastable zone. The "X" points on the plot are boiling points of saturated syrups at 20 cm. Hg. Abs.

Figure 1



One device, currently being marketed, might well solve this problem. It utilizes two temperature measurements; that of vapor leaving the boiling surface and the temperature of hot water or condensate flashing over a second matched bulb at the existing absolute pressure. An electronic bridge system computes the difference between the two temperatures and combines it with the water temperature to give an output indication reading directly in oversaturation. Even sugar boilers with minimum experience have been quickly taught to boil consistently superb sugar using the device even in the face of varying absolute pressure.

GOOD PRACTICE RULES

It seems unthinkable that anyone would contest the premise that pan floor operation could be improved in every mill and refinery and beet sugar factory in the world. Sugar is being produced but there is much spinning of wheels, excessive recycling of syrups, poor use of available equipment, wasted time and money. This minor essay has tried to point out some of the goals of sugar boiling, some of the principles that point the way to achieving them and ways to use these principles to gain immediate benefit from their application.

An equally important subject is that of pan floor and syrup management but a discussion of that must be deferred. When good sugar can be consistently boiled, it will be time enough to get into further operational improvements.

Every good high yield strike boiled on a pan floor reduces the circulating load, providing more time to do better work on all other pans. This can take the route of boiling at somewhat lower and safer oversaturation values for better grain formation or reducing the volume of seed used so as to produce larger, more easily purged grain from the lower purity pans. Time and again, pan floors frantically dropping poor low yield massecuites to keep ahead of syrup supply have been miraculously cleared out by just boiling a few good high yield strikes of finished sugar. In one instance, a beet sugar factory was in the habit of dropping sloppy, low yield white strikes because there was not much head from the mixer to the centrifugal goose necks. A high consistency, high yield white strike was dropped and the world fell in because it took over 25 seconds to fill each centrifugal basket as against the normal 10 seconds. But the mixer was empty long before the succeeding strike because of the fact that each basket stayed filled almost to the outside edge instead of shrinking in to a much lower load. Two such strikes convinced the centrifugal station operators that they could indeed cut out a heavy strike in less time but the remarkable thing was that two good high yield strikes decimated the syrup supply to the point that the sugar boilers had to wait for a graining charge. The sugar was going out to storage instead of recirculating.

In another instance, a large beet sugar factory was having trouble drying their sugar and the trouble was easily traced to the fact that the sugar boilers were being allowed to exceed safe oversaturation values during final concentration. False grain adhering to the otherwise good crystals was roughening the faces, interfering with drying and creating a dust problem. When the factory superintendent was convinced that he would be better off to let the excess high purity liquor overflow to the high raw pan feed tanks; he would not boil any more liquor because it was all going there anyway and at least he would solve the drying and dust problem, he issued an order to stay within oversaturation limits during final concentrating of the white pans and, magically after two or three white pans of clean grain were dropped, there was no further hysteria; the white pan liquor supply was hardly adequate for a graining charge.

Every little good practice rule of sugar boiling adds something to the overall results. It might not be out of order to recapitulate those presented here, aiming first at finished sugar strikes since they are the ones that can get sugar out of the boiling house instead of recirculating.

Graining level. Keep it low to gain on initial agitation and to give maximum time for level increase to maximum so that grain can be grown at minimum oversaturation. Safety and better grain formation are the rewards.

Seeding. Give it a chance to win and be reproducible. Wet ground seems to be the best; set standardized grinding procedures and adhere to them. Periodically adjust slurry quantity to keep desired M.A. of final sugar. If dry hammermilled fondant must be used, disperse it in suitable media with maximum agitation to break up joined grains to minimize seeding of potential conglomerates.

Oversaturation at seeding. To minimize conglomerate grain formation, syrups should be seeded in the 30 to 50% oversaturation zone and held there for a matter of 10 minutes or so until crystals have passed the conglomerating stage. Lower oversaturations discourage conglomeration but require increased seed dosage because more small seed are lost before they are able to survive in the variable oversaturation zones of a vacuum pan. The seeding and holding procedure is a compromise but it is easily resolved in any plant after a few trial runs.

Initial grain growth. Once crystals have passed the stage where conglomeration is important, oversaturation can be increased to a value just short of spontaneous nucleation. Grain grows rapidly but safely.

Boiling consistency. Rapidly growing grain soon begins to occupy more of the massecuite volume and as it comes toward 15% yield, begins to influence the viscosity over and above that of the syrup itself. This effect is often referred to as the strike "coming together". If high oversaturations are held after this point, overall consistencies increase beyond the point of good fluidity and massecuite circulation. It is better then to feed incoming syrups on a basis of consistency and let oversaturation fall away in preparation for final concentration to dropping consistency.

Final tightening. Watch oversaturation carefully. If it approaches the upper limit, feed some water or cut steam flow. A few more minutes spent at this stage are money in the bank. Drop at the highest consistency that can be handled.

Curing. Hold as long as possible in the mixer before spinning to get additional grain growth.

General. If pans get ahead of syrup supply, slow down the boiling rate to prolong strikes. It will make better grain and safer oversaturations can be carried.

Feed Syrups. Maintain them at a temperature high enough to preclude the possibility of crystallization. Syrups, hot enough to flash on entering a pan add to massecuite circulation and this is always desirable.

IS BETTER BOILING WORTH WHILE ?

Indeed it is. By simply following the few good practice rules listed above, pan floors have been able to dramatically reduce the circulating syrup load. In one beet sugar factory some years ago, oversaturation indicators were installed on the pans to guide the sugar boilers and they were taught to follow good procedures on every strike. The pan floor which had always been overloaded and which was the bottleneck of factory production began to operate at a very leisurely pace. The total amount of solids being processed on the pan floor dropped by almost 40%, molasses purity decreased, sugar quality improved and the beet slice rate was increased. The pan floor handled the additional load with ease. This is not an isolated case by any means but was one on which results were carefully checked. Many other plants have reported large improvement in operation and economy after instituting similar upgrading of pan floor techniques.

Sugar boiling is just a rather specialized crystallization process. Once the myths and old wives tales surrounding it have been exposed to the light of day and discredited, constructive improvements to the process become apparent and are easily effected.

(1) Foster, D. H. and P. G. Wright (1962) Control of Crystallization in Vacuum Pans. Proc. I.S.S.C.T., 11:940-950.
(2) Rogers, T. and C. Lewis (1964) Further Studies in the Improve meat of White Sugar Quality in Vacuum Pans. Br. Sug. Corp. Ltd. Tech.Conf.,17:1-31
(3) Kamoda, M. and T. Yamane (1959) On the Formation of Conglomerates of Sugar Crystals. Proc. Res. Soc. Jap. Sugar Ref. Tech., 8:123-130.