Laboratory Equipment for Low Raw Operations

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It is often desirable to determine the effects of process changes on raw side operations, and the effects of such changes are generally difficult to determine in the factory itself. The principal questions to be answered are: What effect does the process change have on the purity of molasses and what is the effect on quality of raw sugar? Experience has shown that even factory trial does not always give the final answer because other variable factors may overbalance the effect of the one under trial. Hungerford, in January 1942, presented before this group data showing seasonal changes in crystallization rates of sugar from beet syrups, the changes being sufficient to mask completely the effects of process treatments being made in the factory during the periods represented by the samples he studied. Therefore it is desirable to have a laboratory procedure capable of giving a fair idea of what the effects of the syrup treatment would be in actual factory operations. The great advantage of the laboratory trial lies in the ability to compare liquors prepared by standard practice and by the new practice, from the identical beets, and thus eliminate the primary faults of factory trials.

The equipment consists of a pan, a crystallizer, and a laboratory centrifuge. The pan, with a capacity of 3,750 grams of dry matter, lias a conical steam-jacketed basal section, and an inner sheet metal shell around which circulation develops. The pan is provided with thermometers for measuring massecuite temperature, a device for accurate control of pressure within the pan, a surface condenser, and a large burette for measuring the volume of the condensate. Pan liquor enters at the bottom and is weighed in from a balance.

The crystallizer is a modified 2-quart ice cream freezer. It is driven by a motor and reducing gears at 0.8 r.p.m. The mixing device of the freezer has been modified in two respects. The wooden scarcer blades have been shaved off sufficiently to clear completely the can wall during rotation, so that grinding of sugar crystals is prevented. And the stirrer mechanism has been so shortened that it may be completely submerged by the massecuite, thus preventing incorporation of air during the cooling process. The top point of the shoulders which carry the wooden scrapers is $4^{1/4}$ inches above the bottom of the can, and the can is filled with massecuite to a point

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about 1/2-inch above these shoulders. The stirrer mechanism rests on a washer.

Temperature control of cooling is maintained by circulation of water, held in a 5O-gallon wooden barrel, around the can containing the massecuite. The water supply, heated to 70° C. at the time the massecuile is placed in 1he crystalizcr, is permitted to cool spontaneously overnight, during which period the temperature drops to slightly below 40° C.

The centrifuge is a standard laboratory type machine, carrying an 8-inch basket.

A little practice with the equipment above described enables one to obtain very satisfactory results in duplicate tests. However, an accurate interpretation of the results requires an accurate knowledge of the properties of the liquors boiled.

The most important factor in the handling of the raws is the preparation of the pan liquor. The purities of the pan liquors must be accurately established, and since variations in juice or syrup treatments may cause changes in the polarization of non-sugars present, only enzyme hydrolysis may be depended upon in the sucrose analysis. Careful drying on sand is required for the dry substance detertions between true and apparent sucrose and dry substance may be employed 1o establish factors, and subsequent analyses may consist of apparent purities and refrectometer dry substances.

The solubility of sugar in the liquors handled should be known, since *ii* is generally necessary that comparative tests on different syrups be made by boiling at the same supersaturation.

Various investigators have published data on solubilities of sugar in impure solutions. Solubilities at constant purity and temperature vary decidedly in the raw pan purity range. If one is studying the effect of juice treatments which do not cause any appreciable effect on the composition of the impurities, he knows that the solubilities of sugars in the liquors is constant at constant purity and an accurate knowledge of solubility is unnecessary. It is only necessary that liquors be boiled under constant conditions of supersaturation at equivalent stages in the boiling process, and that the supersaturation be not sufficiently high to cause formation of false grain. But if treatments of liquors cause appreciable change in the composition of impurities, then solubility determinations on the differently prepared liquors are required. The shape of the solubility curves has been sufficiently well established that a single determination of solubility of sugar in the syrup in question somewhere within the range of 70 to 75 purity at about 70° C. suffices to yield the information necessary to permit one to calculate the concentrations required to

produce the desired degree of super-saturation while boiling in the pan.

However, many raw pan studies do not demand absolutely accurate knowledge of solubilities and in the work in this laboratory the data published in Ind. and Eng. Chem., Vol. 20, p. 1230 (Nov. 1928) and Vol. *25*, p. *555* (May 1933) are generally used as the basis for calculations of concentrations at the various supersaturations.

In addition to a knowledge of solubilities, a knowledge of boiling points of solutions at varing purities and pressures is required. Such data have been published in the literature, but in this laboratory we employ unpublished data we have obtained.

With a knowledge of solubilities and boiling points, one is prepared to calculate a schedule of boiling. For low raw pans we have found a supersaturation of 1.04 to be very satisfactory.

$$\text{(Supersaturatioii} = \frac{\text{(Percentage dry substance in saturated pan liquor)}}{\text{(Percentage dry substance in pan liquor)}}$$

We have also standardized on boiling at 70° C.

Calculations are made in advance to enable the operator to control the rate of drop in purity of the mother liquor, and as the purity drops the pan pressure is decreased to maintain the supersaturation at constant boiling temperature.

The boiling schedule shows the conditions which should exist at any instant during the boiling period. These conditions are: Quantity of pan liquor introduced; quantity of condensate removed; purity of mother liquor; pressure of boiling; and temperature, which remains constant.

It has been pointed out that a sugar solution of given purity and supersaturatioii can boil at the same temperature and pressure as another of higher purity and higher supersaturatioii, and therefore the boiling schedule may not reflect accurately the conditions within the pan. However, tests on mother liquor from the massecuite at the time the pan is dropped show a remarkably close agreement between actual and calculated purities, and furthermore, if conditions within the pan are continuously maintained on schedule, no opportunity is given for purities and supersaturations to depart from calculated values.

For preparation of boiling schedules we have standardized on 76.0 purity pan liquor at 70.0 dry substance, boiled at 70° C Experience has shown that pan liquors should be carefully filtered before use or insoluble matter may completely upset results in the centrifuge. A graining charge of 1,200 grams of dry substance is brought to the supersaturatioii at which the pan is to be boiled and 20 grams of starch-free powdered sugar, suspended in saturated sugar solution,

are introduced through the feed line. The pan is then boiled according to the calculated schedule. At the end it will generally be found necessary to remove carefully some water, after all feed has been charged, since a mass dropped at the super-saturation at which boiling has been done will usually be of too low concentration to produce molasses of low purify. The equipment described will produce 60 purity molasses from a massecuite of about 91.5 percent dry substance.

After the massecuite is dropped from the pan, the crystallizer can is filled to the proper point and crystallization is permitted to proceed for 22 hours. Shortly before spinning, the massecuite temperature is raised to 40° C.

The centrifuge, basket and casing as well, are heated to a little above 40 C. One kilogram of massecuite is placed in the basket, which carries a standard sugar screen, and the massecuite is spun for a standard time at standard speed, say 3,000 r.p.m., for 10 minutes. The unwashed raw sugar is taken for analysis. Since the standard sugar screen is generally loosely fitting and permits the passage of an undue quantity of sugar into the mother liquor, the molasses sample is obtained by spinning a second batch on a finer, more closely fitting screen. The product so obtained is not crystal free but closely simulates molasses produced in factory operations.

The results in table 1 are given as an example of results obtainable with the above described equipment and procedure. The performances of five different liquors were compared.

Table 1.—Experimental boiling of raw massecuite. Pan liquor—purities 76.0, boiled at 1.04 supersaturation, in crystallizer 22 hours.

Liquor No.	Raw mas Dry substance	Molasses True purity	Raw sugar Apparent purity
1	....	58.7	89.0
1	91.8	59.0	92.6
2	....	59.1	90.2
2	91.0	59.1	92.5
3	91.3	60.2	93.1
3	91.8	60.1	92.8
4	92.1	60.1	87.8
4	91.2	61.1	91.9
5	91.4	60.9	91.9
5	91.8	61.1	92.3

Only on Liquor Xo. 4 did molasses purities fail to check, and this was evidently the result of too great variations in raw massecuite concentrations. Two of the massecuite dry substance samples were lost, and in both instances it appeared that the massecuites were slightly too heavy. Small variations in massecuite concentration obviously affect raw sugar purities much more than they do molasses purities.