# VAISALA

### Cane sugar syrup Cane sugar crystallization | 1.02.04



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### **Measured medium**

Sugar and sweeteners

#### **Typical end products**

 Sugar and syrup for sweetening soft drinks, beer brewing, preserves,

beverage, sweets, liqueurs, ethanol, etc.

### Introduction

Crystallization is the final stage in a cane sugar mill and it refers to the formation of the sugar crystals from the concentrated syrup.

Crystallization takes place in vacuum boiling pans. These vacuum pans are the heart of the sugar manufacturing plants. The efficiency of the factory, the quality of the sugar product, the capacity of the plant and the thermal balance, all depend on the operation and control of the vacuum pans to produce a high quality massecuite.

### Application

The *thick juice or syrup* is fed to the vacuum pans and evaporated until saturated. Crystallization is initiated by adding (or seeding) fine sugar crystals to the pan, or *strike*. The crystals start to grow, and the process is continued until the crystals reach the specified size.

The resultant mixture is known as *massecuite* (raw sugar crystals and molasses). The sugar crystals are separated from the molasses by centrifugation and are then washed with hot water to remove any adhering syrup.

### **Control of crystallization**

Crystallization can only take place if the solution is *supersaturated*. Supersaturation is a multivariable function of several parameters of the liquid phase only (syrup or mother liquor). Crystals can grow only if supersaturation is higher than 1.0.

No single instrument can provide on-line data on supersaturation. Conventional sensors used to monitor crystallization provide data of a single massecuite parameter only. However, two sensors can provide information on the important massecuite parameters. These sensors are not influenced by other process parameters:

- Vaisala's process refractometer. The refractive index technology is successfully used for selective concentration measurement of the liquid phase over the complete crystallization strike.
- 2. A sensor for measurement of the total solids content (brix of the massecuite).

Control of supersaturation is critical to the final outcome of the strike. The number of crystals should remain constant from the seeding until the end of the strike. If the supersaturation drops outside its limit, the crystals will stop growing and might even melt. If the supersaturation level rises too high, new crystals will form spontaneously. Spontaneous nucleation results in poor quality crystals of irregular shape and size (fines and conglomerates) which require reprocessing.

## Instrumentation and installation

Zutora SeedMaster-4 is a unique, fourth generation crystallization transmitter and seeding device to be used with Vaisala's process refractometer. The SeedMaster-4 allows for accurate in-line and realtime monitoring of supersaturation and crystal content over the complete process of crystallization, and implementation and control of automatic or manual seeding. The SeedMaster-4 can be connected to one or two process refractometers and to one or two crystallizers.

The SeedMaster-4 provides the following:

- 1. Electronic data capture on massecuite parameters.
- 2. On-line calculation and transmission of massecuite parameters for the advanced control of sugar crystallization with control system.
- 3. Organization and storage of strike history data archive.
- 4. Advanced communication with the control system.
- 5. Automatic seeding of the vacuum pans.
- 6. Serves as user interface for the pan and control system operators.

The refractometer is installed directly into the crystallizer. The measurement is accurate and reliable and the prism remains clean because of crystal friction. Typical measurement range in this application is 65-90 Brix.

Control of crystallization with the refractometer increases productivity, reduces the need for crystal re-processing and guarantees a high-product quality.

Instrumentation	Description				
	SeedMaster-4 for multiparameter sugar crystallizaton monitoring and automatic seeding. Crystallization transmitter and seeding device is used with the Process Refractometer PR-23-GP. It allows for accurate in-line and real-time monitoring of supersaturation and crystal content over the complete strike, and implementation and control of automatic or manual seeding.				
	The Vaisala Polaris PR53GP Process Refractometer is a heavy-duty instrument with non- weld body construction for diverse applications. The optional flange-mounted pipe flow cell installation accessory allows flange-mounted installation in a wide variety of pipe sizes.				
Prism wash system with warm water	For applications with sticky process conditions the prism wash options are steam wash system, pressure wash system ja water wash system.				
Measurement range	Refractive Index (nD) 1.3200-1.5300, corresponding to 0-100 Brix.				
Chemical curve	R.I. per BRIX at Ref. Temp. of 20 °C				

### Beet sugar, cane sugar

### Sugar crystallization: massecuite solids content and mother liquor concentration



### Introduction

This note explains the methods and calibration procedure for measuring successfully the massecuite solids content and mother liquor concentration in sugar vacuum pans over the entire crystallization process. Both parameters need to be measured and controlled, as they influence the quality of the sugar crystals.

Massecuite solids content, or total sugar content, is typically determined using e.g. microwave measuring technology whereas mother liquor concentration (dissolved sugar) is measured with a refractometer. The common measurement scale is Brix.

### Refractometer

Vaisala Polaris<sup>™</sup> Process Refractometer is successfully used for selective measurement of liquid phase over the complete crystallization strike. Due to the unique digital principle, the refractometer measures the true concentration of the mother liquor, without being influenced by the sugar crystals or bubbles in the pan. Moreover, the refractometer does not require re-calibration.

# Massecuite solids content meter

A microwave sensor can measure only the total solids (liquid and undissolved solids phase). Microwave probes are based on the measurement of attenuation and phase shift of microwave radiation. Both are related to the length travelled by the radiated signal, and the density and dielectric characteristics of the medium.

Phase shift is the result of decreasing speed of propagation. Due to the fact that water has a high dielectric constant compared to sugar and the accompanying non-sugars, the water content (and, consequently, solids content) is the major parameter, which determines the dielectric properties of the medium. As an output the microwave probes provide density or solids content of the massecuite.

# Particularities of the crystallization process

Varying process conditions present a challenge to measuring massecuite solids content accurately. Process medium changes during different production phases from liquid to massecuite and consists of both liquid and crystals. Generally, calibrating the massecuite solids content meter can be quite easy either for the liquid phase or for massecuite phase, but not for both. This means that the massecuite solids content sensors cannot solely produce reliable results as they do not cover the whole processing range.

For accurate results, the calibration must cover the full range from pure liquid to the point where Vol. 55 % of the massecuite is crystals. However, if the vacuum pan is also equipped with a refractometer, the calibration difficulties can be mostly avoided.

In the beginning of the strike the process medium is pure liquid. At this point the refractometer and the massecuite solids content meter should give the same measurement value (Figure 1).

The crystals are introduced only after the pan has been seeded. After seeding the massecuite solids content increases as the crystal content increases, whereas refractometric concentration stays rather constant (±3 Brix). Improved accuracy in massecuite solids measurement can be achieved when a refractometer is combined with a microware sensor. Moreover, a refractometer can offer calibration reference value for the massecuite solid content meter at the seeding point.

The recommended control practice is to use a refractometer to measure the concentration from the beginning of the strike until the seeding point. The refractometer provides accurate and repeatable seeding.

### **Calibration procedure**

The process refractometer is factory calibrated according to International Commission for Uniform Methods of Sugar Analysis (ICUMSA) Brix table. The factory calibration should be verified against the production laboratory when commissioning the instrument. The laboratory reference values should be taken in the beginning of the strike when there is only liquid present in the vacuum pan. Only a small BIAS adjustment might be needed to match the refractometer and the production laboratory.



Figure 1. Refractometer and massecuite solids content measurement trends before and after calibration. The liquid concentration and massecuite solids content should be the same before seeding. After seeding they separate as the crystals start to grow.

Typically, the massecuite solids content meter needs regular calibration and calibration checks. The best calibration result is typically achieved when the microwave density meter is calibrated on a narrow range for either liquid sugar or massecuite. In vacuum pans the recommended calibration practice is to calibrate the microwave density meter from seeding point to the end of the strike, which means that the reference samples should be taken at the seeding point, end of the strike and one sample in between (minimum three samples).

Sample	LAB (Mass.Sol.Cont)	Refractometer (Concentration)	Microwave (Mass.Sol.Cont)	Difference refractometer -LAB	Difference microwave/nuclear -LAB
1	77.6	77.5	78.5	-0.1	+0.9
2	79.5 (seeding)	79.4	80.9	-0.1	+1.4
3	85.6	78.5	86.3	-	+0.7
4	90.5	77.9	91.3	-	+0.8

#### Table 1. Example of calibration table.

Table 2. Example of calibration table after calibration procedure. Refractometer offset adjustment +0.1 Massecuite solids meter offset adjustment -0.9.

Sample	LAB (Mass.Sol.Cont)	Refractometer (Concentration)	Microwave (Mass.Sol.Cont)	Difference refractometer -LAB	Difference microwave/nuclear -LAB
1	77.6	77.6	77.6	0.0	0.0
2	79.5 (seeding)	79.5	80	0.0	+0.5
3	85.6	78.5	85.4	-	-0.2
4	90.5	77.9	90.4	_	-0.1



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