AN 5424
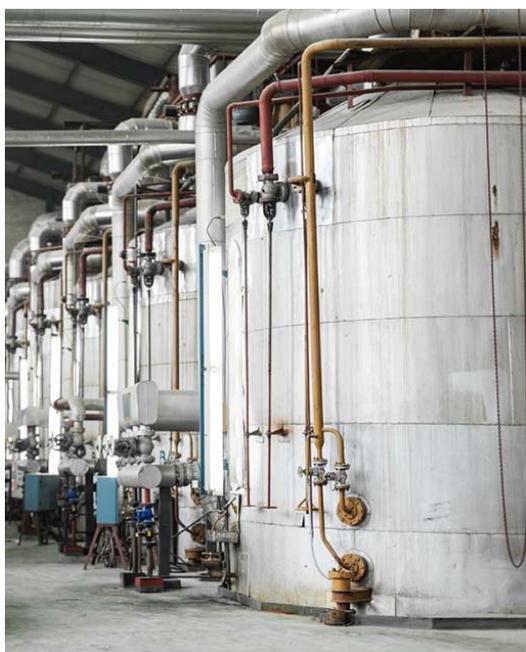
Rev. 2

NIRS™ DS2500

Massecuite

Massecuites are classified according to purity as A, B, or C massecuites. Massecuite is made from supersaturated syrup with sugar crystals added to it, which starts the crystallisation process. Usually massecuite B-seed crystals are used for the A-pan and C-seed crystals for the B-pan.

A-massecuite is utilizing a syrup with a purity from 83 - 88 %. After about 60 % of the sucrose have been crystalized, the massecuite total solds are around 92 %, close to solid. If the total solids are higher than this, the crystals cannot be separated from the molasses during centrifugation and the fluidity of the massecuite is decreased.

The A-molasses is sent to a B-massecuite pan. In the B-massecuite, the amount of sugar crystals cannot exceed around 50 % and to run the crystallisation until a specified target point is crucial. A quick measurement aids the operators that will no longer need to wait 30 or 60 minutes for lab results. The molasses from the B-massecuite is sent to the C-massecuite for further crystallization, but the C-crystals cannot be used as marketable sugar. The crystals are too small and the purity is low. The C-massecuite crystals can be used in feed formulations or, more commonly, the "seed magma" is returned as seeds to the B-massecuite.

This application is suitable to both conventional- and diffuser mills. Dilution of the sample is not necessary and chemicals like dry lead or Octapol are not required. After inserting a sample of massecuite into an NIRS™ DS2500, Pol and Moisture are simultaneously analysed in less than a minute.
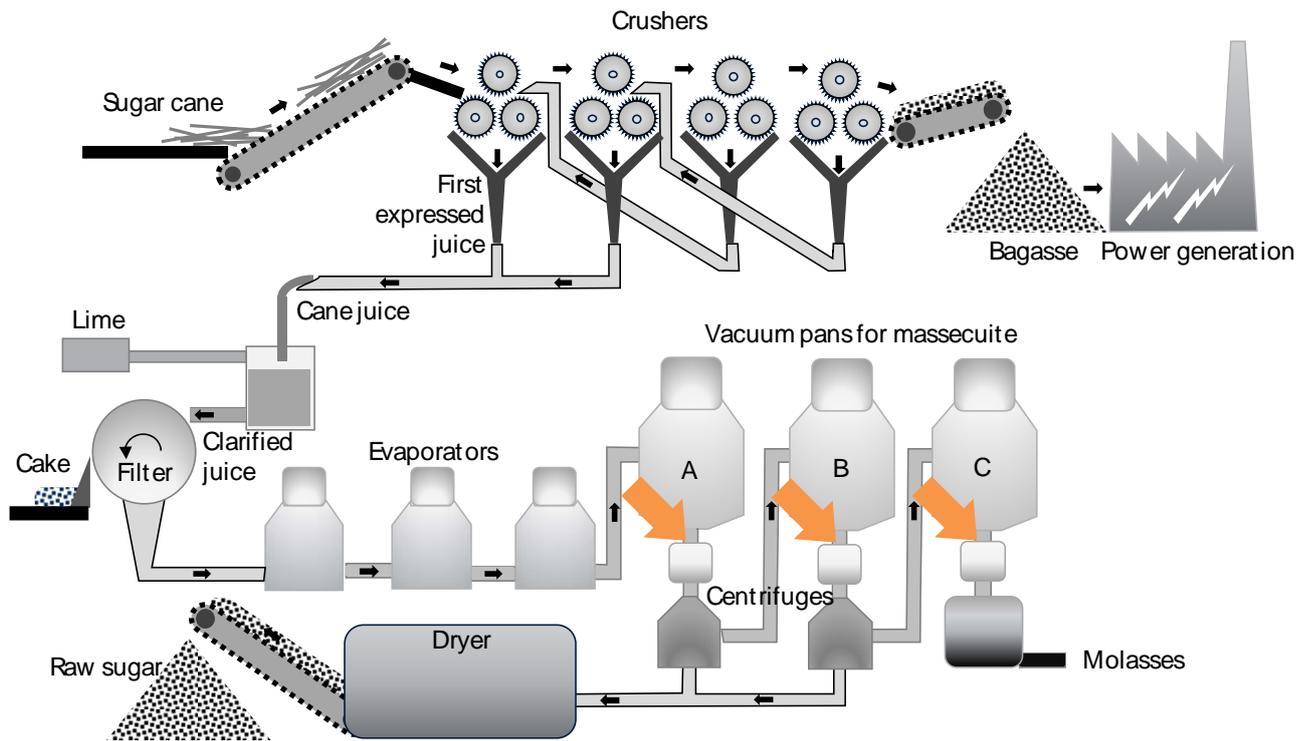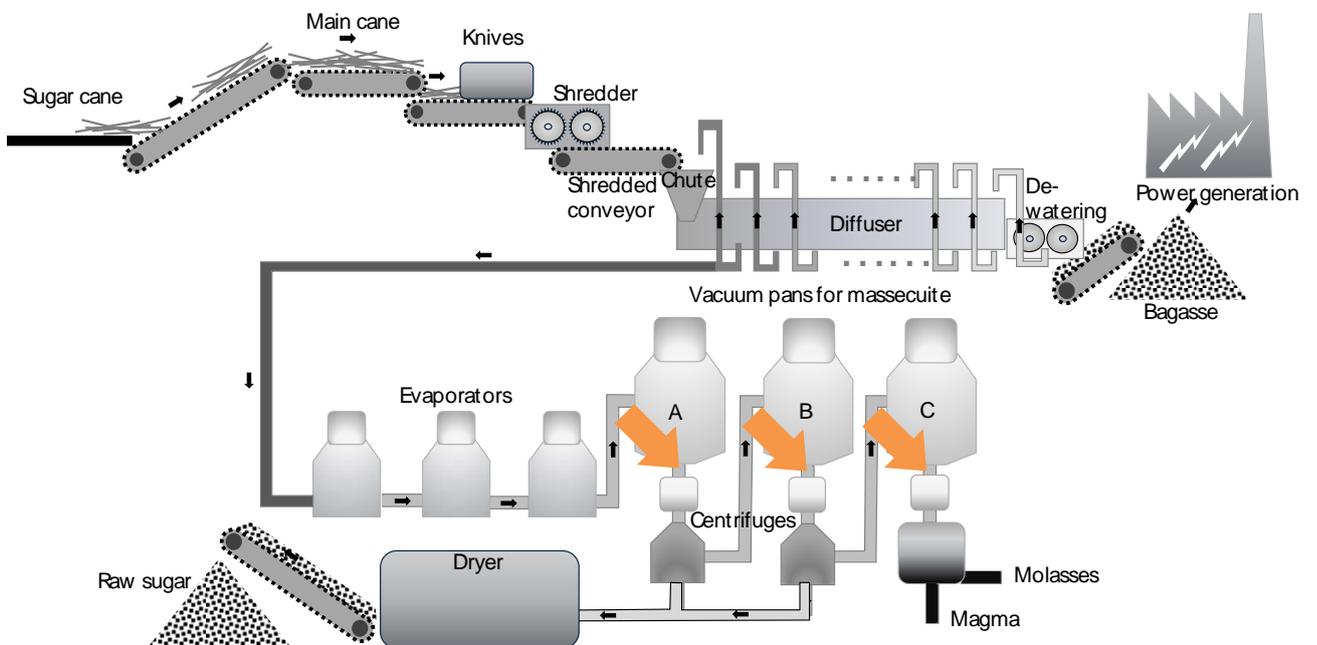
Fig. 1   Measurement points.

Fig. 2   Diffuser mill, measurement points.

Fig. 3   NIRS DS2500

Sample Description

Reference samples have been collected and analysed over several crushing seasons.

Parameter	Version	Min	Max	N	Model type
Brix	2.0.0.0	80.8	95.8	1667	MPLS
Pol	2.0.0.0	52.6	84.5	1662	MPLS
Reducing sugars	2.0.0.0	1.6	9.1	388	MPLS

Table 1   Calibration data.

Performance

Validation statistics is based on samples that were not in the calibration set.

Parameter	Min	Max	N	SEP	RSQ
Brix	88.8	95.2	94	0.52	0.860
Pol	53.7	81.1	85	1.39	0.958
Reducing sugars	1.9	9.4	19	0.39	0.973
Min.: Minimum reference value in test set. Max.: Maximum reference value in test set. N: Number of samples in the test set. SEP.: Accuracy of test set expressed as Standard Error of Prediction (SEP). RSQ: Linear correlation between NIRS DS2500 result and reference result					

Table 2   Validation data.
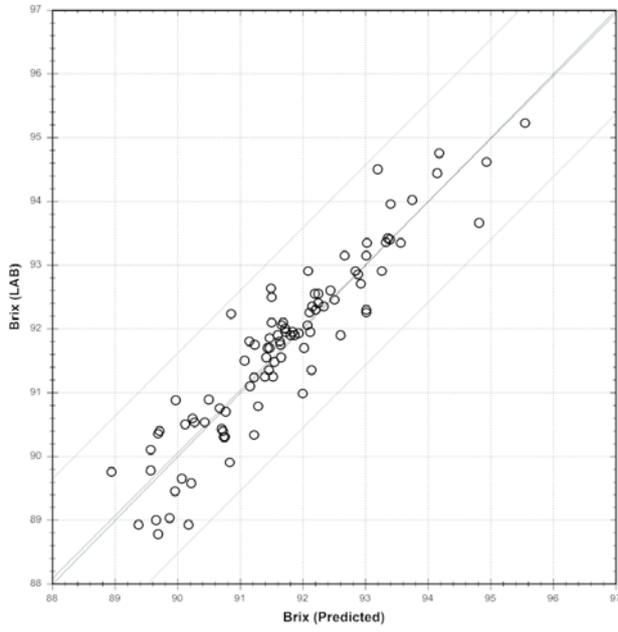
Calibration Performance Graphs
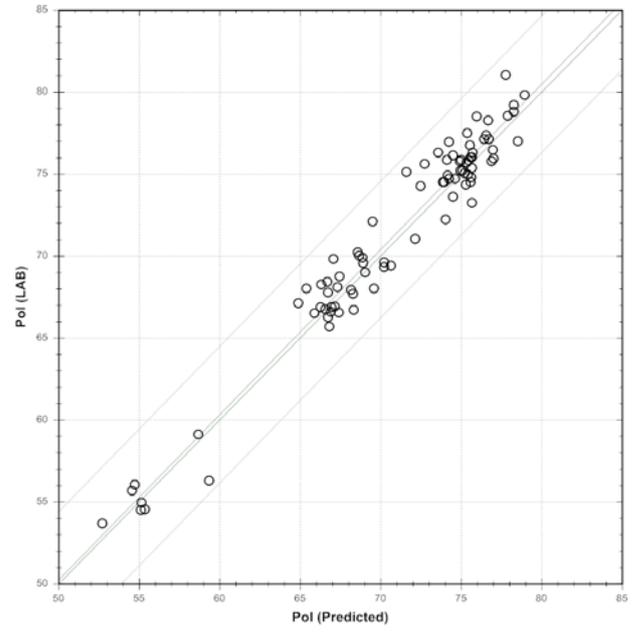

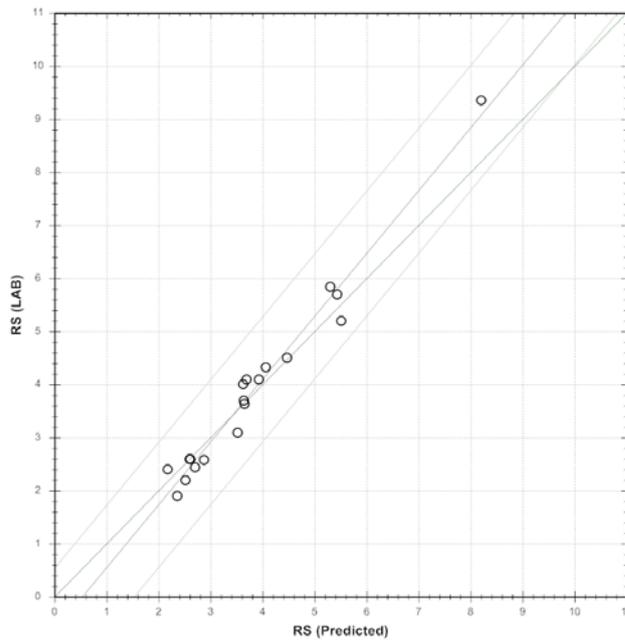
Fig. 4   Brix

Fig. 5   Pol

Fig. 6   Reducing Sugars

In the sugar industry, "Pol" is an abbreviation for Polarisation and it is synonymous with sucrose. "Brix" is synonymous with Total Dissolved Solids. Purity is a key for process optimisations and we strongly recommend to add it as a calculated parameter:

Purity = Pol/Brix*100 %

The amount of Reducing Sugars ("RS") is another quality indicator. RS or "Invert Sugars" consist mainly of glucose and fructose originating from sucrose inversion. The less RS, the better. All four calibrations are built on wide concentration ranges. Massecuite contains sugar crystals and it is a very thick liquid. Sampling errors are therefore contributing to the rather large SEP values of the Pol and the Brix calibrations.

Note:

The performance example outlined in this note should only be regarded as a guideline for the expected performance of new installations. The performance of new installations will always depend on the uniformity of the sample preparation and the homogeneity of the product, as well as the accuracy of the reference method used and the range for the test samples. An indication of the obtainable performance can be found as approximately 1.5 to 2 times the reproducibility of the reference method. If the samples measured exceed the stated calibration ranges, or have non-common variations of other components, this might also influence the performance of the calibrations.

Each sample will be analysed and compared to the calibration database. Three key values will be given as an indicator to how well the unknown sample fit the calibration samples:

- Global H value (GH) - measures how far the spectrum is from the centre of the database. A high GH value corresponds to a sample far from the calibration database, meaning a sample different from the calibration samples. If the GH value exceeds a certain limit, the sample is suspected to be out of the calibration working range.

- Neighbourhood H value (NH) - measures how close the sample is to the nearest sample in the database. A high NH value corresponds to a sample far from the nearest neighbouring sample in the calibration database, meaning a sample different from the calibration samples. If the NH value exceeds a certain limit, the sample is suspected to be out of the calibration working range.

- T-statistics - measures the predicted parameter compared to its calibration range in the database counted as number of standard deviations. A value of zero corresponds to the average of the parameter in the database. A high positive value of more than 3 standard deviations indicates that the predicted value is at the high end or outside the range of the database. A negative value of less than -3 standard deviations indicates that the predicted value being at the low end or outside of what is in database.

Default Warning and Action limits for GH, NH, and T-statistics are set for each prediction model in the software.

Sample Preparation

We recommend using the slurry cup with a 0.5 mm gold reflector for analysis of massecuite. No special temperature stabilisation has been made so it is recommended to analyse the samples at room temperature.

Fig. 7   Massecuite sample in Slurry Cup.

Ordering and Further Information

Please contact Henrik Hansen, Head of Market Innovation, hha@foss.dk.