CORE SAMPLING RECOMMENDED PROCEDURES

Prepared

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CORE SAMPLING

Introduction

The core sampling system for cane quality evaluation and cane payment was introduced in Louisiana in 1976. At present all sugar factories in the state use the core sampling system.

The system comprises the following operations:

1. Use of a corer to obtain a cane sample from the delivery.
2. A sample preparation device to prepare the core sample.
3. A hydraulic press to extract juice from the prepared core sample.
4. The analysis of the press juice for Brix, pol and percent of sediment. The analysis of the press residue (bagasse) for moisture.

The system is capable of yielding results that provide for fair cane payment.

The core data can also be used by factory managements to evaluate factory performance. However, for the core system to achieve its potential, all aspects of the system require routine monitoring and the use of standard methods.

Sampling of Cane

The Louisiana core sampler is of the overhead, inclined core tube type. The inclined core tube was selected because of the use of whole stick harvesters in Louisiana.

The core tube should be set so as to penetrate the delivery to the full depth of the cane in the wagon. The forward motion of the core tube in the delivery should approximate the average stalk length of the cane.

Cane for sampling should be stacked with the cane in the same direction (i.e. all of the tops should face in the same direction). For cane loaded in the same direction, the core tube entry point in the load is immaterial, since regardless of where the core tube enters the delivery a sample along the whole stalk length will be obtained.

Cane should not be loaded “butt-to-butt” or “tops-to-tops”, since depending on the entry point of the corer primarily tops or alternatively primarily bottoms of the cane will be sampled, leading respectively to overestimation or underestimation of the cane quality.

If for any reason, cane is stacked “butt-to-butt” or “tops-to-tops”, the delivery should be positioned so that the corer enters at one end of the stalks. This would yield the most representative sample for this type of delivery.
Routine Maintenance

The corer should be routinely inspected to make sure that:

1. The saws on the tip of the core tube are sharp so that a cutting rather than punching action is obtained.
2. The core ejection plunger retracts fully during the coring operation, and ejects fully during the sample discharging operation.

Sample Preparation

At the end of the coring cycle the corer ejects the cane core into a sample preparation device that prepares and subsamples the core sample for pressing. The J&L Model X-8000 unit is currently used for sample preparation.

The sample preparation device should:

1. Achieve a reasonable degree of cane preparation to facilitate sample homogenization and accurate subsampling.
2. Have very little hold-up (i.e. be self-cleaning) so that farmers’ samples are not commingled.
3. Not dry the cane sample.
4. Be cleaned on a routine basis.

Note that if the sample preparation units are cleaned with water, then at least one core sample should be passed through the unit to absorb the water before a sample for analysis is processed.

The clearances in the units should be maintained within the manufacturers' recommended limits.

Hydraulic Press

The hydraulic press is used to press a 1000 gm sample of prepared cane to yield a juice and residue (bagasse) for analysis.

The presses should operate with a pressure of 3650 psig on the sample. For the 3-hole J&L presses this is equivalent to a hydraulic oil pressure of 3000 psig, while on the single hole presses this pressure is reached with an oil pressure of 2450 psig.

Bottom drainage of the juice from the cylinder is recommended, since this yields the greatest retention of field soil in the bagasse residue. In the case of the J&L Model X-6000 single hole press, a circular piece of high grade centrifugal screen is cut to fit over
the bottom of the cylinder. In the case of the J&L Model X-9000 press, a piece of filter paper is generally placed over the perforated bottom to prevent blockage of the drainage holes.

In the case of the J&L Model X-9000 three-hole press, numbering of the individual holes is recommended to keep track of samples.

Press Cleanliness

Thorough daily cleanings of the press and juice piping is essential. The addition of a few drops of a suitable mill biocide (e.g. Busan 881) to each 1000 gm press charge will help to keep the press free of contamination.

If the press is washed down during normal daily operations, the water remaining in the inaccessible parts of the system should be removed by pressing a core sample to flush the press. No weights or analyses should be performed on this sample as it will be diluted with water.

The collection of juice from the press should be conducted over the entire pressing operation.

Timeliness of Sample Processing

At peak sampling periods, more samples may be collected than can be accommodated by the core lab. The storage of core samples for pressing for extended periods (1-2 hours) can result in sample deterioration. Samples should be processed within two hours of coring.
ANALYTICAL PROCEDURES

The analyses required are routine analyses similar to those performed in the factory lab.

The juice is analyzed for:

1. Brix using a refractometer.
2. Pol using an automatic polariscope.
3. Sediment using a centrifuge.

The bagasse residue is analyzed for:

1. Moisture using an oven.

Sediment Test

The sediment test is carried out on the press juice using a lab centrifuge.

The entire press juice sample as collected from the press should be vigorously stirred to ensure complete suspension of the sediment. The centrifuge tube should then be filled to the mark with the stirred press juice as soon as possible after stirring. If the press juice is not stirred vigorously immediately before sample removal the sediment test will not accurately reflect the true sediment in the juice.

Once the centrifugal tubes have been filled with the juice sample, the tube can be placed in a test tube rack until a full complement of tubes are ready for centrifuging. The centrifuging should be carried out at high speed (1500 G) for 10 minutes.

Note: The centrifuge should always be operated with a balanced load to reduce vibration.

Juice Brix

The Brix determination should be run on a filtered or centrifuged press juice sample.

If the sediment test is performed, the supernatant liquid in the centrifuge tube after centrifuging can be used for the Brix determination. The liquid can be removed with a dropper. The dropper should be dry (as in the case of disposable plastic droppers) or thoroughly rinsed in the juice. If the dropper is reused, it is recommended that the droppers not be washed with water between analyses, but rather that they be flushed several times with the juice sample. A few drops of zero Brix water in the juice will affect the Brix reading much more than a few drops of the juice from a previous sample of comparable Brix.

If the sediment test is not performed, then a suitable Brix sample can be obtained by filtering some of the juice through the same type of filter paper used for the pol
determinations. In this case the same suggestions for the droppers given above are applicable.

The Brix is determined on a refractometer. Some factories use the temperature corrected Brix setting, while others record both the uncorrected Brix and temperature. While the latter method is more correct, the use of the former (temperature corrected Brix setting) introduces only a negligible error and reduces the number of readings required.

**Juice Pol Reading**

Approximately one teaspoon of Horne’s dry lead is added to approximately 250 ml of press juice. For the non-lead method, approximately 1 teaspoon of the ABC or Octopol clarifying agent is added to approximately 250 ml of press juice.

The mixture is vigorously shaken until the dry lead or non-lead clarifier is completely dispersed and then allowed to stand briefly before filtering through a funnel with filter paper into a beaker.

The initial filtrate should be discarded since it is generally cloudy.

The quantity of clarifying agent should be increased or decreased depending on whether the filtrate is dark or cloudy.

When sufficient filtrate has been collected to rinse and fill the 200 mm polariscope tube, the funnel with filter paper can be removed. The filtrate should be used to thoroughly rinse the pol tube twice before the pol tube is filled. The pol tube is then placed in the polariscope and read. The pol reading should not blink (Optical Activity polariscopes) nor should the booster light come on (Rudolph IIS polariscopes). If either of the above occur this indicates a cloudy juice or the presence of a bubble in the pol tube. Cloudy juice can usually be cleared by refiltering or by rerunning the sample. As a last resort cloudy samples can be cleared by adding one drop of acetic acid to the juice in the pol tube and mixing by inverting and rocking the pol tube.

**Moisture % Residue**

The moisture content of the residue is determined by drying a sample of the residue to constant weight in convection ovens at 150 deg. C.

The entire press residue is crumbled by hand and thoroughly mixed. Approximately 50 grams of the mixed press residue is weighed into an aluminum pan or other suitable container. The weighed moisture samples can be left out until an oven load of samples is ready for drying. The samples for drying are placed in a convection oven leaving some space between the containers and the oven sides to provide good air circulation. The timer for timing the drying time is started as soon as the oven door is closed. After sufficient drying to ensure that constant weight has been achieved, the samples are removed from the oven and reweighed. The loss in sample weight on drying divided by the initial sample weight expressed as a percent is the moisture content of the residue.
APPENDIX

CORE SAMPLING CALCULATION METHOD

The following data is required:

- Residue Weight % Cane (by weighing)
- Moisture % Residue (by weighing)
- Extracted Juice Brix (by refractometer)
- Extracted Juice Pol (by polariscope)
- Sediment Volume % Juice (by centrifuging)

**Sediment Correction**

The sediment volume % juice is multiplied by a factor to convert the sediment volume % juice to dry sediment weight % juice:

\[
\text{Dry Sediment % Juice} = \text{Sediment Volume % Juice} \times \text{Factor}
\]

The factor being used is 0.302.

The grams of dry sediment in the juice is given:

\[
\text{Dry Sediment in Juice} = \text{Juice Weight} \times \text{Dry Sediment % Juice} / 100
\]

The juice weight is taken as the cane weight charged to the press (1000 gm) less the residue weight.

**Corrected Residue**

Had the sediment stayed in the residue, instead of getting into the juice, then the additional residue weight would have been:

\[
\text{Extra Residue} = \text{Dry Sediment Weight} / (1 – \text{Moisture % Residue}/100)
\]

This extra residue is added to the weighted residue from the press and used to calculate the residue % cane for use in the core calculations.

**Fiber % Cane**

Assuming that the extracted juice and residual juice have the same composition the fiber % residue is given by:

\[
\text{Fiber % Residue} = 100 - \frac{\text{Moisture % Residue}}{(1 – \text{Extracted Juice Brix}/100)}
\]
Thus the fiber % cane, F, is given by

\[
\text{Fiber} \% \text{ Cane} = (\text{Fiber} \% \text{ Residue}) \times (\text{Residue} \% \text{ Cane}) / 100
\]

And the absolute juice is given by

\[
\text{Absolute Juice} \% \text{ Cane} = 100 - \text{Fiber} \% \text{ Cane}
\]

**Brix % Cane and Pol % Cane**

The Brix % Cane can be calculated as follows:

\[
\text{Brix} \% \text{ Cane} = B = (\text{Juice} \% \text{ Cane}) \times (\text{Brix} \% \text{ Juice}) / 100
\]

The Pol % Cane can be calculated as follows:

\[
\text{Pol} \% \text{ Cane} = P = (\text{Juice} \% \text{ Cane}) \times (\text{Pol} \% \text{ Juice}) / 100
\]

The above expressions allow the Brix, Pol and Fiber % Cane to be calculated.

The sugar yield can be obtained from:

\[
\text{Theoretical Sugar Yield, lbs 96 sugar/gross ton of cane} = \frac{2000 \text{ lbs} \times \text{Pol} \% \text{ Cane} \times \text{Extraction} \times \text{Retention} \times \frac{1}{100} \times \frac{1}{100} \times \frac{1}{100} \times 0.96}{\text{ton}}
\]

The extraction is obtained assuming a constant reduced extraction with an absolute juice lost % fiber of 56.67, thus the pol extraction is:

\[
\text{Pol Extraction} = 100 - 56.67 \times (\text{Fiber} \% \text{ Cane}) \times (100 - \text{Fiber} \% \text{ Cane})
\]

This expression for the pol extraction predicts pol extractions that depend on the fiber content of the cane, for example:

<table>
<thead>
<tr>
<th>Fiber % Cane</th>
<th>Pol Extraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
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</tr>
<tr>
<td>10</td>
<td>93.70</td>
</tr>
<tr>
<td>12.5</td>
<td>91.90</td>
</tr>
<tr>
<td>15</td>
<td>90.02</td>
</tr>
<tr>
<td>20</td>
<td>85.86</td>
</tr>
</tbody>
</table>
The retention is obtained using the Winter-Carp formula and multiplying by the Boiling House Efficiency (BHE):

\[
\text{Retention} = (1.4 - \frac{40}{\text{Extracted Juice Purity}}) \times \text{BHE}
\]

**Current Corer TRS Prediction (1975-1997)**

Substituting the reduced extraction expression developed above for the pol extraction, the Winter-Carp formula for the boiling house retention, and assuming a boiling house efficiency of 96, the sugar yield expression reduces to:

\[
\text{TRS} = (0.28P - 0.08B) \times \frac{(100 - 56.67F)}{100-F}
\]

where
- \(\text{TRS} = \text{Theoretical Recoverable Sugar, lbs 96 sugar/ton cane}\)
- \(P = \text{Pol \% Cane}\)
- \(B = \text{Brix \% Cane}\)
- \(F = \text{Fiber \% Cane}\)

**New (1998- ) TRS Prediction**

Using the fibraque correction, the following calculations should be used:

- \(\text{New Fiber} = \text{NF} = F \times 1.3\)
- \(\text{New Pol} = \text{NP} = P \times \frac{(100-\text{NF})/(100-F)}\)
- \(\text{New Brix} = \text{NB} = B \times \frac{(100-\text{NF})/(100-F)} \times Z\)

where \(Z = 1.15 - 0.0018(1000 - \text{Corrected Residue Wt.}) / 10\)

\[
\text{TRS} = (0.28\text{NP} - 0.08\text{NB}) \times \frac{(100 - 56.67\text{NF})}{100-\text{NF}}
\]

where
- \(\text{TRS} = \text{Theoretical Recoverable Sugar, lbs 96 sugar/ton cane}\)
- \(\text{NP} = \text{Pol \% Cane}\)
- \(\text{NB} = \text{Brix \% Cane}\)
- \(\text{NF} = \text{Fiber \% Cane}\)

The Liquidation Factor = \text{Actual Factory Sugar Production, lbs 96 sugar} / \text{Total lbs TRS Calculated for All Cane}
The Commercial Recoverable Sugar, CRS = TRS x Liquidation Factor

The new absolute juice analysis is given by:

\[
\text{Brix} = \frac{\text{NB}}{100 - \text{NF}} \times 100
\]

\[
\text{Pol} = \frac{\text{NP}}{100 - \text{NF}} \times 100
\]

\[
\text{Purity} = \frac{\text{NP}}{\text{NB}} \times 100
\]
RECOMMENDED LABORATORY EQUIPMENT, CHEMICALS AND SUPPLIES

Equipment/Instruments
Polariscope (automatic digital type) with 200 mm pol tubes and quartz plate
Refractometer (temperature compensated, digital type)
Balances (Digital electronic, taring type) with check weights
Lab Centrifuge (and accessories)
Ovens (with mercury in glass thermometers)
Surge suppressors or constant voltage transformers – for use with balances, refractometers and polariscopes

Chemicals/Reagents
ABC or Octopol or other non-lead clarifying agent
Acetic Acid
Biocide
Detergent
Distilled Water
Ethanol
Filter Cel
Hydrochloric Acid
Lead Sub-Acetate (Horne’s dry lead)
Sucrose

Supplies
Beakers – sugar type glass beakers, or clear disposable plastic (10 oz. Solo cups)
Bags – plastic, for cored cane samples and press residue cakes
Bottles/Containers – for collection of press juice
Bottle Brush
Jars/Bottles – for juice pol analysis
Bowl/container – for weighing cane and residue samples
Centrifuge tubes – plastic, graduated (15 or 25 ml)
Data Sheets
Filter Paper – dry and good quality (Bartlett 226 or equivalent)
Foil Pans – for residue moisture determination
Funnels – Urbanti funnels, plastic and ribbed, glass or copper ribbed sugar funnels
Plunger – for removal of residue from cylinder after pressing (if necessary)
Labels/tape
Oven mitts/Potholders – for handling hot moisture containers
Pens/pencils/markers
Pipets – disposable plastic (DisPo pipets or equivalent) for refractometer
Soft tissue/Kleenex – for wiping refractometer prism
Spoons/stirring rods
Squirt bottle –for distilled water
Stoppers – for jars/bottles
Timers – for ovens and centrifuge
Thermometers – for ovens
Towels/paper napkins
Volumetric Flask – 100 or 200 ml
LABORATORY PROCEDURES

Hydraulic Press

Operating Pressure

The two most common hydraulic presses are the J&L X-6000 single hole press and the J&L X-9000 three hole press.

The X-6000 press has an oil piston diameter of 7” and a sample piston of 5.687” diameter. The ratio of piston areas is 1.5151. Thus with an oil pressure of 2450 psig, the pressure on the cane sample is 3712 psig (2450 x 1.5151).

The X-9000 press has an oil piston diameter of 8” and a sample piston of 7.25” diameter. The ratio of piston areas is 1.2176. Thus with an oil pressure of 3000 psig, the pressure on the cane sample is 3653 psig (3000 x 1.2176).

It is recommended that the X-6000 press be operated with an oil pressure of 2450 psig, and the X-9000 press with an oil pressure of 3000 psig. Other presses should be operated so as to produce a pressure on the cane sample of about 3700 psig.

Pressing Time

The normal (and factory set) pressing time (under pressure) for the X-9000 press is 2 minutes. With the 2 minute pressing time the cycle time is about 2:45 min., yielding about 22 samples per hour.

The recommended pressing time for the X-6000 press is also 2 minutes. The turnaround time on the X-6000 press may be longer than for the X-9000 press, and some factories find it necessary to operate at a 1:30 min. pressing time to achieve the desired number of samples per day. Since most of the juice is extracted very early in the pressing cycle, the results from pressing for 1:30 min. or for 2:00 min. will be very similar.

Press Cleaning

The press should not be cleaned with water between samples, since this will dilute the juice. Instead, a few drops of a suitable mill biocide (e.g. Busan 881) should be added to the 1000 gm cane sample for pressing.

If water washing of the press is necessary, some prepared cane should be pressed to displace the water left in the press piping. No analyses should be performed on this sample.

The press should be thoroughly cleaned at the end of each day.
Centrifuge

IEC (International Equipment Co.) Clinical Centrifuge Model 428. Maximum Speed 3000 rpm (1470 G) – radius 14.6 cm.

Dynac Centrifuge. Maximum speed 5500 rpm – diameter 15”.

The centrifuges are operated with 15 or 25 ml graduated centrifuge tubes. The tubes should be filled with the press juice as it comes from the press. The press juice should be vigorously stirred immediately before filling the centrifuge tubes. The press juice should be vigorously stirred so that the centrifuge sample contains a representative quantity of sediment.

The centrifuge should be operated at 1500 G for 10 minutes. The use of a timer is recommended for timing the spinning time.

The centrifuge tubes should be clean and dry.

The centrifuge should be operated with a full complement of tubes, or a balanced load of centrifuge tubes.

Refractometer

American Optical – manual setting, digital readout with temperature, Brix and temperature corrected Brix settings.

Bellingham & Stanley RFM-80 – automatic with same features as above.

Atago Palette 100 – Inexpensive ($1100). Provides temperature-corrected Brix readings only, with a range of 0 to 32 Brix. Battery or AC adapter powered.

Refractometers should be calibrated in accordance with manufacturers’ instructions. After calibration, refractometers should be checked at 0 and 20 Brix using distilled water and a 20 Brix sucrose solution respectively. A 20 Brix sucrose solution is prepared by making up 21.62 grams of sucrose to 100 ml of solution with distilled water in a volumetric flask.

Periodically during the day, the refractometer zero should be checked with distilled water.

The refractometer prism should be wiped and dried with a soft tissue between readings. The juice should be placed on the prism with a plastic dropper. The prism will be scratched and damaged if glass droppers come in contact with the prism.

The juice sample placed on the refractometer should be either filtered press juice or the clear supernatant centrifuged liquid in the centrifuge tube from the sediment test.
**Polariscope**

Optical Activity – automatic digital (1 or 2 decimal place model).

Rudolph Autopol IIS – automatic digital (2 decimal place).

The polariscope should be calibrated according to the directions in the instrument manual.

The polariscope should be zeroed (with reset button), and checked with a quartz plate periodically.

The polariscope tube (200 mm) should be filled with distilled water and checked. Suitable polariscope tubes should read zero with distilled water. Polariscope tubes with over tightened ends will not read zero. If the polariscope tube does not read zero with distilled water it should be fitted with new ends and retested. Pol tubes that do not read zero with distilled water can be used, provided that the polariscope is zeroed with the water filled pol tube in place and provided that the pol tube is always inserted into the polariscope in the same direction.

**Moisture Determination**

The residue moisture samples are weighed on a digital electronic balance with tare capability. A balance with 250 to 500 gm capacity is suitable. A 50 gram sample of the crumbled and mixed residue is weighed into the drying pan.

The forced draft ovens should be set at 150 deg. C using a mercury in glass thermometer. The oven temperature should be set with the oven empty and with the air vents in their normal half open position. Once the wet residue samples are placed in the oven, the oven temperature will fall, but the oven’s thermostat should not be readjusted.

The samples should be dried until constant weight is achieved. The actual drying time necessary will depend on the type of oven and the sample, and will have to be determined by test at each factory.

The ovens should be charged with a full load of samples, and the oven should not be opened until the end of the drying time.

The residue samples should be placed so that there is some space (about ½”) between the sample containers and between the sample containers and the walls. If the sample containers are placed in contact with each other or the wall the air flow will be restricted. After drying for the full drying time, the moisture samples are removed and reweighed. The moisture is calculated from the loss in weight on drying divided by the initial sample weight.

**Acknowledgement**

The core sampling project was funded by a grant from the American Sugar Cane League.
## SAMPLE DATA AND RESULTS

### CALCULATING CORER RESULTS

<table>
<thead>
<tr>
<th>SAMPLE NO.</th>
<th>WT. RESIDUE</th>
<th>JUICE BRIX</th>
<th>POL %</th>
<th>CORR RES</th>
<th>CORR'D. RES WT</th>
<th>JUICE POL</th>
<th>JUICE PUR.</th>
<th>CANE FIBER</th>
<th>CANE FIBER</th>
<th>CANE BRIX</th>
<th>CANE POL</th>
<th>TRS CANE FIBER</th>
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<th>POL NEW CANE FIBER</th>
<th>TRS NEW CANE FIBER</th>
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14