CHAPTER 5
Sampling

1. General

An analysis can be no better than the representivity of the sample and good sampling technique must therefore be applied to ensure that the sample represents the material from which it is taken in an unbiased manner. The nature and importance of the material will govern the sampling specification as to size and frequency. It is easier to obtain a small representative sample if the material is homogeneous, like clarified juice, than if it is a product of mixed composition like cane. Where great accuracy is not required, catch samples taken at fairly large time intervals will often be adequate, particularly when the variability in the composition of the material to be sampled is relatively small. However, where great accuracy is required, continuous sampling should be employed and care should be taken that the ratio between the mass of the fraction of the sample extracted in each unit of time and the mass of the material it represents, is constant.

It is sometimes better to take frequent catch samples than a continuous sample, for example, it is desirable to obtain information on the variability of the composition of the material. Catch samples are also taken when there is possibility that a continuous sample will deteriorate during the sample period.

Cleanliness of sampling devices and sample receptacles is essential. This calls for a planned and regular programme of cleaning. In this regard duplicate sets of samplers and receptacles will greatly facilitate cleaning operations and must be provided wherever possible. Sample receptacles should be seamless and constructed of stainless steel or copper and must be covered to minimise evaporation and contamination.

The importance of mixing the primary sample before sub-sampling cannot be over stressed and details are given below, under the procedures described, for the various products.

2. Cane

2.1 Prepared cane for direct analysis

2.1.1 General

For cane payment purposes, individual cane consignments are sampled and therefore there is a need for accurate identification of consignments up to the sample point.

When the first mill is by-passed for mechanical reasons, cane sampling must continue and the miller shall provide the equipment and labour necessary for transferring the excess cane sample back to process.

Apparatus: Electronic cane tracker
Cane sub-sampler
Sample table
Sample shredder
Enamelled billy can with lid (seamless construction and 6 litre capacity)

2.1.2 Procedure for personnel operating the electronic cane tracker

(a) Visually follow the progress of the consignment on the carrier system up to the point where the electronic cane tracker is designed to take over.

(b) Make allowance as dictated by local conditions for intermixed cane so as to ensure that this will not be sampled. Apart from this the sampling period should correspond with the passage of the maximum percentage of a consignment. Special circumstances, e.g. group testing, may dictate that one sample will be taken to represent the cane from more than one supplier, but this will be done only on specific instructions from the Cane Testing Services Manager.

(c) Once all intermixed cane has passed the cane tracker starting point, cause the electronic cane tracker to follow the progress of the consignment as described in Chapter 4, Section 1.

(d) Complete the weighbridge ticket with details required and consign the ticket to the laboratory. Enter the relevant details (cane tracker number and, if applicable, bundle numbers), via the keyboard.

(e) As the end of a consignment passes the cane tracking initiating point, depress the stop button.

(f) After every new consignment check that the tracker output counter number plus the number of consignments in transit, equal the tracker input counter number. If these figures do not agree, immediately investigate the reason and take the necessary measures.

(g) If for any reason mixing of cane from different suppliers occurs after the head of a consignment has passed the cane tracking initiating point, tracking must be interrupted so as to avoid sampling the intermixed portion. When this occurs, one consignment will be shown as two on the display and sampling staff must be advised immediately what action to take. Such incidents must always be recorded.

(h) Check frequently that the tracker output counter corresponds with the sample point counter.

2.1.3 Procedure for personnel operating at sample point

(a) On receiving the cane tracker signal (the green light signal and bell) to commence sampling, place the clean sample receptacle in the appropriate position.

(b) Note the size of the consignment as shown on the cane tracker display and adjust the cane sub-sampler secondary stage reject interval timer so as to obtain the optimum size sample (10 kg) in the sample receptacle.

(c) Fill in a sample ticket showing the sample point counter number and the receptacle number.

(d) After a factory maintenance stop reject the cane from the first two cane sampler openings as a precaution against the possible presence of water in the sampling equipment.

(e) At the end of the sample run which is indicated by the red signalling light and bell, tip the contents of the sample receptacle onto the table provided.
Note: All subsequent cane sampling operations must be done as thoroughly and quickly as possible to avoid selective sampling and loss of moisture.

(f) Mix the cane and then spread into a layer 50-70 mm thick. By randomly taking handfuls of cane collect one sub-sample of about 2 kg.
Care must be taken not to shake the cane held in the hand as this will result in particle size selection.

(g) Discard the excess sample on the mixing table.

(h) Transfer the sub-sample to the sample shredder disturbing the sample evenly in the shredder compartment.

(i) After shredding as described in Chapter 4, Section 7 transfer the sample from the shredder sample receiver to the enamelled billy can, taking the precaution of first

wiping out the billy can with a portion of the cane sample and rejecting this cane portion. At some installations a pneumatic sample conveyor system has been installed. In these instances transfer the sample from the shredder sample receiver to the plastic bag provided. Close the bag by tying a knot at the open end and prick the plastic a few times to release trapped air. Fit the plastic bag into the canvas carrier bag which is attached to a pneumatic sample conveyor shuttle.

(j) Immediately convey the sample in the closed billy can with relevant sample ticket to the laboratory, or alternatively, where pneumatic sample conveyors are in operation, place the shuttle with carrier bag attached in the air tube system.

(k) The work area as well as all equipment must be maintained in a clean state at all times.

3. Final bagasse

3.1 General

Because of the difficulties of continuous sampling of bagasse, catch samples are taken at regular intervals. Different types and configurations of bagasse conveyors at mills have given rise to various methods for obtaining a sample representative of the full depth and width of the final bagasse blanket. Ideally sampling should be through a hatch situated in the base plate of the bagasse elevator just after the last mill. Such a hatch must span the full width of the elevator and open sufficiently to allow the fall-out of a complete slat load of bagasse. Opening and closing of the hatch must be with a snap action to avoid bias.

However, the increasing trend in the South African Industry towards the use of belt conveyors precludes the use of a sample hatch and at mills where this already obtains, use of the "swing" sampler as described in Chapter 4, Section 8 is recommended.

Apparatus: Hatch sampler or, if not applicable, swing sampler
Sample receptacle – enamelled billy can with lid, seamless construction (6 litre capacity)
Mixing table (stainless steel top)

3.2 Procedure

(a) Regardless of chokes or other irregularities of crushing, a sample of bagasse should be taken at a predetermined time every hour. If the mill is not crushing at the sampling time, no sample shall be taken for that hour.

(b) If the hatch sampler is used, the hatch is opened so as to allow a full slat load of bagasse to fall through. Reject this sample and collect a second sample for analysis.

(c) If the swing sampler is used the sampling procedure is as follows:
(i) Remove the locking pin and swing the handle down until it rests on the guide wheel.
(ii) Push the sample box into the bagasse stream ensuring that the leading edge moves beyond the rear of the falling bagasse stream and then reverse the stroke.

(d) The hatch sample is mixed on the sample table while that obtained with the swing sampler is mixed in the sampler box.

(e) In either case random handfuls which are placed in the sample receptacle, are taken to provide a sub-sample of *ca.* 1 kg. In sub-sampling by hand, care must be taken not to shake the bagasse held in the hand as this will result in a biased sample.

(f) Immediately convey the bagasse sample in the closed sample receptacle to the laboratory.

4. Mixed juice

4.1 Pol, Brix and sucrose

Apparatus: Mixed juice sampler (with chiller for hot juice)
Sample receptacle - seamless stainless steel bucket with lid (15 litre) (with immersion cooler for hot juice)
Juice mixer
Bottle - wide mouth, with lid (450 cm^3)
Measuring cylinder (50 cm^3)
Polythene tubing (100 micron wall thickness; the tubing is flattened at manufacture to give a width of 75 mm)
Heat sealer
Alcohol bath
Deep freezer (-40°C)

Reagent: Juice preservative

4.1.1 Procedure

(a) The sample must be collected continuously over the hour.

(b) At the commencement of the hour, add juice preservative to the clean dry sample receptacle (0.2 cm^3 preservative per litre of juice to be collected) and position the receptacle with lid at the sampler outlet.

(c) At the end of the hour remove the sample receptacle at the same time as the scale reading is taken and replace with a second receptacle, previously cleaned and dried and to which the requisite quantity of juice preservative has subsequently been added.

(d) The receptacle containing the juice is kept covered with the lid and immediately conveyed to the laboratory.

(e) In the laboratory thoroughly mix the juice in the receptacle using the juice mixer.

(f) Transfer a portion of the juice into a 450 cm^3 bottle taking the precaution of first rinsing the bottle with a portion of the juice and discarding the rinsings.

(g) The samples for pol, Brix and sucrose analyses are drawn from the 450 cm^3 bottle. For sucrose analysis, however, hourly samples are not analysed individually, but analysis is conducted instead on a weekly composite sample prepared as described in steps *(h)* to *(n)* below.

(h) Cut a 125 mm length of the plastic tubing and double seal one end using the strip heat sealer.

(i) Rinse the measuring cylinder with a portion of the mixed juice (discard the rinsings) and then measure out an aliquot of approximately 20 cm^3 but proportional to the tonnage of the mixed juice recorded for the hour.

(j) Pour the juice from the measuring cylinder into the plastic sachet.

(k) Double seal the sachet approximately 40 mm from the open end, taking care to expel as much of the air as possible before sealing.

(l) Label and seal the remaining 40 mm portion of the sachet in accordance with the requirements of the CTS laboratory.

(m) Place the sachet in the alcohol bath stored in the deep freezer so as to ensure rapid freezing.

(n) After 10 minutes in the alcohol bath transfer the sachet with frozen mixed juice sample to the storage container in the deep freezer.

(o) Repeat steps *(h)* to *(n)* throughout the week for each hourly mixed juice sample.

(p) At the end of the week the frozen sachets are despatched to the SMRI in the insulated containers provided for this purpose.

4.1.2 Procedure for thawing frozen mixed juice samples

The operation is carried out at the SMRI laboratory.

(a) Remove the frozen sachets from the container and hang them on the special racks provided.

(b) Immerse the rack of frozen samples in the ambient temperature running water bath for 10 minutes.

(c) Remove the rack from the water bath, allow the excess water to drain and immerse the rack with samples in the alcohol bath for a few seconds.

(d) Remove the rack with samples from the alcohol bath and stand for 20 minutes in the air chamber (ambient temperature) so that the sachets are dried by a forced draught of air.

(e) Remove the sachets from the rack and place on a clean sheet of paper towelling. Cover with a second sheet of paper towelling and gently press so as to absorb any remaining moisture/alcohol.

(f) Inspect for any evidence of juice leaks and discard any sachets found to be leaking.

(g) Cut open the sachets and expel the contents into a clean dry 6 litre billy can.

(h) Once all the juice has been transferred from the sachets to the billy can, thoroughly stir the composite sample and place the lid on the billy can.

4.2 Insoluble solids determination

4.2.1 General

Representative sampling of mixed juice for insoluble solids requires proper sampler design and positioning in view of the propensity for heavy insoluble solids to segregate and not be dispersed uniformly through the juice flow.

Apparatus: Cutter sampler
Seamless stainless steel bucket with lid (12 litre)

Reagent: Juice preservative

4.2.2 *Procedure*

(a) Sampling is conducted over the shift such that each hour two cuts (forward and back) are taken across the full cross section of the juice flow.

(b) The sampler delivers approximately 5 litres of juice over the shift at the cut frequency specified in *(a)* above.

(c) Just before the beginning of the shift take the clean dry sample bucket and add 1 cm^3 of juice preservative (0.2 cm^3 per litre of sample.)

(d) At the beginning of the shift, place the sample bucket with lid beneath the sampler outlet. The outflow from the sampler is led through the lid opening and onto the receptacle by means of a length of polythene tubing.

(e) Depress the control valve which activates the sampler to move fully across and then back again through the juice stream.

(f) Repeat step *(e)* each hour throughout the shift.

At the end of the shift remove the sample receptacle with the sample for analysis and replace with a clean, dry receptacle to which 1 cm^3 of juice preservative has subsequently been added.

(h) All the sample in the sample receptacle must be taken to the laboratory for sub-sampling; do not discard any if the sample is larger than usual.

Note: If for some reason the sample receptacle is found to have filled to overflowing the whole sample must be rejected as it will be biased.

(i) In the laboratory the sample is cooled if necessary prior to sub-sampling and analysis.

5. Clarifier mud

5.1 Pol and Brix

Apparatus: Sample receptacle - enamelled seamless billy can with lid (3 litre)
Plastic bottle - wide mouth with screw top
Beaker (250 cm^3, graduated)

5.1.1 Procedure

(a) Take a catch sample once an hour using the clean dry billy can.

(b) Take the sample from across the mud flow as it discharges from the weigh tank of the scale.

(c) Put the lid on the billy can and convey the sample to the laboratory.

(d) In the laboratory cool the sample to ambient by standing in a water trough at room temperature for approximately 30 minutes.

(e) When the sample has attained ambient temperature remove the can from the trough and dry the exterior.

(f) Rapidly stir the sample in the billy can and using the beaker transfer 100 cm^3 of the agitated sample to the plastic bottle.

(g) Store the bottle with sample in the deep freeze.

(h) Repeat steps *(a)* to *(g)* each hour for four hours.

(i) At the end of the fourth hour place the plastic bottle with the 4-hour composite sample in a water bath and thaw the frozen sample as rapidly as possible to 20°C ± 2°C.

(j) Remove the bottle from the water bath, dry the exterior and proceed to analyse the sample for Pol and Brix immediately.

5.2 Insoluble solids

5.2.1 General

Sampling for insoluble solids is only required in circumstances of partial re-routing or intermittent (within the same week) re-routing of clarifier muds.

Apparatus: Sample receptacle – enamelled seamless billy can with lid (3 litre)
Bottle – wide mouth with glass stopper (2 litre)
Beaker (250 cm^3, graduated)

5.2.2 Procedure

(a) Take a catch sample once an hour using the clean dry billy can.

(b) Take the sample from across the mud flow as it discharges from the pipe outflow into the mud scale header tank.

(c) Put the lid on the billy can and convey the sample to the laboratory.

(d) In the laboratory cool the sample to ambient by standing in a water trough at room temperature for approximately 30 minutes.

(e) When the sample has attained ambient temperature remove the can from the trough and dry the exterior.

(f) Add 0.3 cm^3 juice preservative to the clean dry bottle.

(g) Rapidly stir the sample in the billy can and using the beaker transfer 150 cm^3 of the agitated sample to the plastic bottle.

(h) Repeat steps *(a)* to *(g)* each hour until the end of the 8-hour shift.

6. Filter feed (mud)

6.1 **Pol and insoluble solids** (press water clarifier mud only)

6.1.1 General

When press water clarifier mud is not returned to the mixed juice but instead is weighed and then sent to the filters, it is necessary for cane payment and factory control purposes to determine the Pol and insoluble solids content of the mud.

Apparatus: Sample receptacle - enamelled seamless billy can with lid (3 litre)
Bottle - wide mouth with glass stopper (2 litre)
Beaker (250 cm^3, graduated)

6.1.2 Procedure

(a) Take a catch sample once an hour using the clean dry billy can.

(b) Take the sample from across the mud flow as it discharges from the weigh tank of the scale.

(c) Put the lid on the billy can and convey the sample to the laboratory.

(d) In the laboratory cool the sample to ambient by standing in a water trough at room temperature for approximately 30 minutes.

(e) When the sample has attained ambient temperature remove the can from the trough and dry the exterior.

(f) Add 0.3 cm^3 juice preservative to the clean dry bottle.

(g) Rapidly stir the sample in the billy can and using the beaker transfer 150 cm^3 of the agitated sample to the bottle.

(h) Repeat steps *(a)* to *(g)* each hour until the end of the 8-hour shift.