Method 4.6 – filtrate and filter feed: filter retention of mud solids

1. Rationale

This method is applicable to filtrate and filter feed and determines the filter retention of mud solids from filter feed to filtrate expressed on Brix.

2. Principle

The Brix of a filtrate and a filter feed sample is determined. The bagacillo from fresh samples of filtrate and filter feed is removed through screening. The mud solids of the screened samples are determined gravimetrically by filtration and drying of the residue. The mud solids on Brix in the filtrate sample is expressed as a percentage of the mud solids on Brix in the filter feed sample to give the percentage filter retention.

3. Apparatus

3.1 **Top pan balance** readable to 0.01 g

3.2 **Heavy duty balance** readable to 1 g

3.3 **Drying oven** operating at 130°C

3.4 **Stainless steel container**: 5 litres

3.5 **Beakers**: 250 and 500 cm^3

3.6 **Conical flask** with stopper: 1 000 cm^3

3.7 **Screen**: 200 mm ϕ, 0.075 mm pore opening

3.8 **Buchner funnel**: 110 mm ϕ

3.9 **Buchner flask**: 500 cm^3

3.10 **Glass rod**

3.11 **Desiccator** containing self-indicating silica gel

3.12 **Filter paper**

Whatman No. 6, Postslip medium white w/s or equivalent (for Brix): 185 mm ϕ
Whatman No. 1 or equivalent (for mud solids): 150 mm ϕ

3.13 **Refractometer** operating at 20.0 ± 0.1°C

3.14 **Schott bottle**: 250 cm^3

3.15 **Filtration apparatus**

funnels: 2 × 100 mm ϕ stemless

beakers: 4 × 250 cm^3
watch/cover glasses: 2 × 100 mm ϕ

4. Reagents

4.1 Celite 577 filter aid

Celite is an inert powder and inhalation may cause asbestosis of the lungs. Wear a dust mask during use.

5. Procedure

5.1 Brix in filtrate and filter feed

Pipette 50 cm^3 of the well-mixed sample and transfer to the 250 cm^3 Schott bottle. Weigh 1 g Celite 577 powder while wearing a dust mask and add to the Schott bottle. Mix and filter the solution through fluted filter paper (Whatman No. 6, Postslip medium white w/s or equivalent) supported in a funnel which rests directly on a beaker. Seal the funnel with a watch glass to minimise evaporation. Discard the first 10 cm^3 of filtrate and collect about 20 cm^3 of the filtrate in another clean, dry beaker. Do not allow the filtrate to touch the bottom of the funnel or filter paper. Do not replenish the solution in the filter funnel.

Zero the refractometer using distilled water.

Pour the filtrate into the refractometer cell compartment using three portions to ensure complete displacement of the previous solution. Record the reading once it stabilizes at 20.0°C.

5.2 Mud solids in filtrate and filter feed

Weigh the 5 litre container on the heavy duty balance and record the mass. Dry the Whatman No. 1 filter paper at 130°C in the oven for 2 hours. Store in a desiccator until needed.

Stir the sample vigorously to ensure that insoluble matter is uniformly dispersed throughout. Quickly weigh a sub-sample of the agitated sample in a beaker according to the masses indicated in Table 1.

Table 1: Sub-sample and aliquot amounts of filtrate and filter feed

Sample	Mass sub-sample (g)	Mass aliquot (g)	Volume of beaker (cm^3)
Filtrate	500.0	500.0	1000
Filter feed	100.0	250.0	250 and 500

Transfer the sample to the screen and wash with distilled water until there is no trace of mud left in the bagacillo. Collect the filtrate and all washings in the 5 litre container. Place the 5 litre container on the heavy duty balance and add distilled water until the mass of the contents is 2 000 g.

Weigh the filter paper on the top pan balance within 30 seconds after removing the paper from the desiccator. Wet the filter paper in a Buchner funnel with distilled water so that the paper fits tightly around the sides of the funnel. Mix 2 g of Celite 577 with 20 cm^3 of water and pour down a glass rod onto the filter paper while low vacuum is applied to precoat the filter paper with a thin layer of Celite. Use a 15 kPa differential pressure.

Stir the contents of the 5 litre container and quickly weigh an aliquot while stirring in a beaker according to the masses indicated in Table 1. Pour this aliquot slowly down a glass rod onto the precoated filter paper at a rate slower than the drainage rate of the filter so as not to flood the filter aid surface. This will ensure quick filtration. Stir the contents of the beaker occasionally while filtering. Visually inspect the filtrate for clarity to ensure that no filter aid or suspended solids are passing through the filter. Use a 15 kPa differential pressure.

Rinse the beaker with 10 × 30 cm^3 distilled water and pour each rinse through the filter paper, allowing the filter to drain between washings. Release the vacuum and transfer the filter paper and contents to a preweighed 250 cm^3 beaker. Dry the beaker and contents in the oven at 130°C for 4 hours. Cool in a desiccator for 1 hour and weigh.

6. Calculations

6.1 Brix in filtrate and filter feed

Correct the refractometer reading for the water blank.

6.2 Mud solids in filtrate and filter feed

$$\text{Mass of sample} = \frac{\text{mass of sample (g)}}{\text{total mass of dilution (g)}} \times \text{aliquot (g)}$$

$$\text{Mass of mud solids (g)} = M_2 - M_1$$

where M_1 ≡ mass of beaker, filter paper and filter aid (g)
M_2 ≡ mass of beaker, filter paper, filter aid and mud solids after drying (g)

$$\text{Mud solids in sample} = \frac{\text{mass of mud solids (g)}}{\text{mass of sample (g)}} \times 100$$

6.3 Filter retention of mud solids

$$\text{Retention (\%)} = 100 - \left(\frac{\text{mud solids in filtrate (\%)}}{\text{mud solids in filter feed (\%)}} \times \frac{\text{Brix in filter feed (\%)}}{\text{Brix in filtrate (\%)}} \times 100 \right)$$

Express as percentage to the nearest unit.

7. Example

7.1 Brix in filtrate

Water blank	=	0.00°
Brix of filtrate at 20.0°C	=	8.59°Bx

7.2 Brix in filter feed

Water blank	=	0.00°
Brix of the filter feed at 20.0°C	=	10.57°Bx

7.3 Mud solids in filtrate

$$\text{Mass of filtrate} = \frac{500\ \text{g}}{2000\ \text{g}} \times 500\ \text{g}$$
$$= 125\ \text{g}$$

Mass of beaker, filter paper and filter aid = 322.83 g
Mass of beaker, filter paper, filter aid and sample = 323.67 g

Mass of mud solids = (323.67 - 322.83) g
= 0.84 g

$$\text{Mud solids in filtrate} = \frac{0.84\ \text{g}}{125\ \text{g}} \times 100$$
$$= 0.672\%$$

Report as 0.67%

7.4 Mud solids in filter feed

$$\text{Mass of filter feed} = \frac{100.00\ \text{g}}{2000\ \text{g}} \times 250\ \text{g}$$
$$= 12.5\ \text{g}$$

Mass of beaker, filter paper and filter aid = 352.53 g
Mass of beaker, filter paper, filter aid and sample = 352.11 g

Mass of mud solids = (352.53 - 352.11) g
= 0.42 g

$$\text{Mud solids in filter feed} = \frac{0.42\ \text{g}}{12.50\ \text{g}} \times 100$$
$$= 3.36\%$$

Report as 3.36%

7.5 Filter retention

$$\text{Retention (\%)} = 100 - \left(\frac{0.67\%}{3.36\%} \times \frac{10.57\%}{8.59\%} \times 100 \right)$$
$$= 100\% - 24.25\%$$
$$= 75.75\%$$

Report as 76%

8. References

SASTA (1985). *Laboratory Manual for South African Sugar Factories.* 3rd Edition: 268 - 273.

SMRI (1997). Determination of the refractometer Brix in juice. *SMRI Test Methods*, TM005.