Method 5.8 – Syrup: conductivity ash

1. Rationale

This method is applicable to factory syrups. The conductivity ash gives a measure of the concentration of the ionised soluble salts present in the sample with conductivities of up to 500 μS/cm at concentrations of up to 5 g/100 cm^3.

2. Principle

The specific conductivity of a sugar solution at a concentration of 5 g/100 cm^3 or less is determined and compared to the specific conductivity of water. The equivalent ash content of the sample as per convention is calculated by the application of a generic conversion factor.

3. Definitions

3.1 Cell constant

The cell constant of a conductivity meter depends on the area of the two platinum plates and their distance apart. Since both these dimensions are fixed, but vary from one cell to another, it is necessary to determine this constant for each cell and for different types of samples. The constant should be checked once a month or whenever either of these two dimensions may have changed.

4. Apparatus

4.1 **Oven** operating at 105°C

4.2 **Conductivity meter**

4.3 **Volumetric flasks**: 100 and 1000 cm^3

4.4 **Beakers**: 50 and 250 cm^3

4.5 **Analytical balance** readable to 0.0001 g

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

4.7 **Water bath** operating at 20.0 ± 0.2°C

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

4.9 **Filtration apparatus**

funnel: 100 mm ϕ stemless
beakers: 3 × 250 cm^3
watch/cover glass: 100 mm ϕ

4.10 **Conical flask**: 150 cm^3

4.11 Filter paper: Whatman No. 6, Postslip medium white or equivalent (185 mm ϕ)

4.12 Watch glass

5. Reagents

5.1 Purified water

Use twice-distilled or de-ionised water with a specific conductance of less than 2 µS/cm for the preparation of all solutions.

5.2 Potassium chloride (0.01 M)

Dry the potassium chloride (KCl) in an oven at 105°C for 3 hours and cool in a desiccator.

Weigh 745.56 mg of dried potassium chloride, dissolve in water in a 1 000 cm^3 volumetric flask and make to the mark. This solution has a conductivity of 1277.96 µS/cm at 20°C after the specific conductivity of the water has been subtracted.

5.3 Celite 577

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

6. Procedure

6.1 Determination of the cell constant

Place a beaker with 100 cm^3 of the 0.01 M KCl solution in the water bath at 20.0°C for 30 minutes. Cover the beaker with a watch glass to prevent evaporation. Read the conductivity of the 0.01 M KCl solution.

Calculate the cell constant according to 7.1. If the measurements cannot be made at 20.0°C a temperature correction must be applied to the theoretical conductance of the KCl solution according to 7.2. A new cell constant must be determined every month.

6.2 Sample conductivity ash

Prepare a solution by dissolving 5 g of the sample in about 60 cm^3 purified water in a 100 cm^3 volumetric flask. Mix to dissolve and add purified water to just under the mark. Stand the flask in a water bath at 20.0 ± 0.2°C for 30 minutes together with a beaker of purified water (5.1) which will be used as a blank. Make the solution up to the mark.

Rinse the conductivity cell twice with the purified water. Transfer a fresh portion of the purified water to a 50 cm^3 beaker and measure the conductivity at 20.0 ± 0.2°C. Rinse the conductivity cell twice with the sample solution. Transfer a fresh portion of the solution to a 50 cm^3 beaker and measure the conductivity at 20.0 ± 0.2°C. If the readings cannot be taken at 20.0°C a temperature correction must be applied according to 7.5.

6.3 Brix determination

Weigh 20.00 ± 0.05 g of molasses into a 150 cm^3 conical flask. Weigh 80.00 ± 0.05 g of water into the same flask to bring the total mass to 100.00 ± 0.10 g. Stopper and mix thoroughly.

Weigh 1 g Celite 577 powder while wearing a dust mask and gloves and add to 50 cm^3 of the stock solution in a beaker. Mix and filter the solution through fluted filter paper supported in the funnel which rests directly on the 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 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.

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 ± 0.1°C.

7. Calculations

7.1 Cell constant

Deduct the specific conductivity of the purified water from the conductivity of the KCl solution.

$$\text{cell constant (/cm)} = \frac{\text{theoretical conductivity } (m\text{S/cm})}{\text{conductivity of KCl solution } (m\text{S})}$$

7.2 Temperature correction for KCl solution

If the KCl solution is not at 20.0°C the theoretical conductivity of the solution must be adjusted according to the equation below. This adjustment must be made before the cell constant is calculated and is only valid in the range 20 ± 5°C.

$$\text{KCl conductivity at T°C } (\mu\text{S/cm}) = \text{Conductivity at 20°C} \times [1 + 0.021 \times (T - 20)]$$

7.3 Brix

Correct the refractometer reading for the water blank. Multiply the corrected value by 4 (or the dilution factor) to obtain the Brix of the syrup.

7.4 Sample conductivity ash (applicable only to syrups)

$$\text{ash (\%) sample} = (C_s - C_w) \times \text{cell constant} \times 0.0018$$

where C_s ≡ conductivity of the sample
C_w ≡ conductivity of purified water
0.0018 ≡ generic method constant

$$\text{ash (\%) syrup} = \text{ash (\%) sample} \times \frac{\text{Brix}}{100}$$

7.5 Temperature adjustment

The temperature adjustment for the sample reading must be made before adjusting the ash (%) sample for the Brix of the syrup.

$$\text{Conductivity at 20°C } (\mu\text{S}) = \frac{\text{ash (\%) sample}}{[1 + 0.023 \times (T - 20)]}$$

Report as percentage to two decimal places.

8. Example

8.1 Cell constant

The conductivity of the 0.01 M KCl solution at 24.0°C is 1351.29 µS and the specific conductivity of the purified water is 1.17 µS. The theoretical conductivity of the 0.01 M KCl solution at 24.0°C is:

KCl conductivity at 24.0°C = 1277.96 × [1 + 0.021 × (24.0 - 20.0)]
= 1385.31 µS

$$\text{cell constant} = \frac{1385.31\ mS/cm}{1350.12\ mS}$$

= 1.026 /cm

8.2 Brix

Dilution factor:

mass of syrup in stock solution	=	25.19 g
mass of total solution	=	100.02 g
dilution factor	=	3.97

Brix at 20.0°C:

water blank	=	0.00°Bx
Brix of the solution at 20.0°C	=	17.9°Bx
multiply by the dilution factor	=	17.9°Bx × 3.97
Brix of the syrup	=	71.1°Bx

8.3 Sample conductivity ash

conductivity of water at 25.0°C = 1.17 µS
conductivity of sample at 25.0°C = 1279.00 µS

ash (%) sample at 25.0°C = (1279.00 - 1.17) × 1.026 × 0.0018
= 2.3599%

$$\text{ash (\%) syrup at 25.0°C} = 2.3599\% \times \frac{71.1}{100}$$

= 1.678%

$$\text{ash (\%) syrup at 20.0°C} = \frac{1.678\%}{[1 + 0.023 \times (25.0 - 20.0)]}$$

= 1.505%

Report as 1.51%

9. References

SASTA (1985). *Laboratory Manual for South African Sugar Factories*. 3rd Edition: 156 - 157, 281 - 282.

SMRI (1997). Determination of the conductivity ash in syrup. *SMRI Test Methods,* TM012.