Method 8.8 – Refined sugar: conductivity ash

1. Rationale

This method is applicable to cane raw and refined sugars. The conductivity ash gives a measure of the concentration of the ionised soluble salts present in the sample with conductivities of up to 30 μS/cm at concentrations of up to 28 g/100 cm^3.

2. Principle

The specific conductivity of a sugar solution at a concentration of 28 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. Conductivity ash is not directly correlated to gravimetric ash as determined by incineration.

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 at different concentration depending on the type of sample. 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, 500 and 1 000 cm^3

4.4 **Beakers**: 2 × 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 **Watch glass**

5. Reagents

5.1 Purified water

Use twice-distilled or de-ionised water with a conductivity 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 is subtracted.

5.3 Potassium chloride (0.0002 M)

Dilute 10 cm^3 of 0.01 M KCl solution in a 500 cm^3 volumetric flask and make to the mark. This solution has a conductivity of 26.6 ± 0.3 μS/cm at 20°C after the specific conductivity of the water is subtracted.

6. Procedure

6.1 Determination of the cell constant

- Place a beaker with 100 cm^3 of the 0.0002 M KCl solution in the water bath at 20.0°C for 30 minutes.
- Cover the beaker with a watch glass to minimise evaporation.
- Read the conductivity of the 0.0002 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

- Prepare a solution by dissolving 28.00 ± 0.01 g of the refined sugar into a 250 cm^3 beaker.
- Add purified water until the total mass of the sugar plus water equal 100.00 ± 0.01 g.
- Cover the beaker with a watch glass and dissolve the sugar gently using a magnetic stirrer.
- Stand the beaker in a water bath at 20.0 ± 0.2°C for 30 minutes together with a beaker of purified water which will be used as a blank.
- 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 7.4.

7. Calculations

7.1 Cell constant

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

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

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.

$$\text{KCl conductivity at T°C } (\mu\text{S/cm}) = \text{Conductivity at 20.0°C} \times [1 + 0.021 \times (T - 20)]$$

7.3 Sample conductivity ash (applicable only to refined sugar)

$$\text{ash (\%)} = [(C_s \times \text{cell constant}) - (C_w \times \text{cell constant} \times 0.35)] \times 0.0006$$

where C_s ≡ conductivity of the sample (µS)
C_w ≡ conductivity of purified water (µS)
0.35 ≡ water correction factor
0.0006 ≡ generic method constant

7.4 Temperature adjustment for sample reading after calculation of ash (%)

$$\text{Conductivity at 20.0°C } (\mu\text{S}) = \frac{\text{ash (\%)}}{[1 + 0.023 \times (T - 20)]}$$

Express as percentage to three decimal places.

8. Example

8.1 Cell constant

The conductivity of the 0.0002 M KCl solution at 24.0°C is 30.57 µS and the specific conductivity of the purified water is 1.17 µS. The theoretical conductivity of the 0.0002 M KCl solution at 24.0°C is:

$$\begin{aligned}\text{KCl conductivity at 24.0°C} &= 26.6 \times [1 + 0.021 \times (24.0 - 20.0)] \\ &= 28.8\ \mu\text{S}\end{aligned}$$

$$\text{cell constant} = \frac{28.8\ \mu\text{S/cm}}{29.4\ \mu\text{S}} = 0.980\ \text{/cm}$$

8.2 Sample

conductivity for water at 25.0°C = 1.17 µS
conductivity for sample at 25.0°C = 22.86 µS

$$\text{ash (\%) at } 25.0°\text{C} = [(22.86 \times 0.980) - (1.17 \times 0.980 \times 0.35)] \times 0.0006$$

$$= 0.013\%$$

$$\text{ash (\%) at } 20.0°\text{C} = \frac{0.013\%}{[1 + 0.023 \times (25.0 - 20.0)]}$$

$$= 0.0117\%$$

Report as 0.012%

9. Precision

The tolerance associated with the analysis is ± 0.001%.

10. References

ICUMSA (1994). Conductivity ash in refined sugar products. *ICUMSA Methods Book,* Method GS2/3-17.

SASTA (1985). *Laboratory Manual for South African Sugar Factories*. 3rd Edition: 156 - 157, 339 - 340.

SMRI (1997). Determination of the conductivity ash in refined sugar. *SMRI Test Methods,* TM013.