Method 6.4 – C-molasses and C-nutsch: conductivity ash

1. Rationale

This method is applicable to C-molasses only. 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 0.25 g/100 cm^3.

2. Principle

The specific conductivity of the molasses 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. The Brix of the molasses is used in the calculation; dry solids as determined by Karl Fisher or vacuum oven can also be used.

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, 200 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 $\pm$ 0.2°C

4.8 **Refractometer** operating at 20.0 $\pm$ 0.1°C

4.9 **Filtration apparatus**

funnel: 100 mm ϕ stemless
beakers: 3 $\times$ 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 Potassium chloride (0.005 M)

Dilute 500 cm^3 of the 0.01 M KCl solution (5.2) in a 1 000 cm^3 volumetric flask and make to the mark. This solution has a conductivity of 651 μS/cm at 20°C after the specific conductivity of the water has been subtracted.

5.4 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 0.005 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 0.005 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

Weigh 5.00 ± 0.02 g of well-mixed molasses in a 250 cm conical flask. Add 50 - 100 cm^3 purified water and mix well. Transfer to a 200 cm^3 volumetric flask and add purified water to the mark. Pipette 10 cm^3 of the solution into a 100 cm^3 volumetric flask and add 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, 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.

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.

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

7.3 Brix

Correct the refractometer reading for the water blank. Multiply the corrected value by 5 (or the dilution factor) to obtain the Brix of the molasses.

7.4 Sample conductivity ash (applicable only to molasses)

$$\text{conductivity} = \text{conductivity reading} - \text{conductivity of purified water}$$

$$\text{ash (\%)} = \left(16.2 + 0.36 \times \frac{\text{Brix}}{100} \times \frac{\text{mass}}{20}\right) \times \text{conduct} \times \text{cell constant} \times \frac{5}{(\text{mass}/20)} \times 10^{-4}$$

7.5 Temperature adjustment for sample after calculation of ash (%)

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

Report as percentage to two decimal places.

8. Example

8.1 Cell constant

The conductivity of the 0.005 M KCl solution at 24.0°C is 716.17 µS and the specific conductivity of the purified water is 1.17 µS. The theoretical conductivity of the 0.005 M KCl solution at 24.0°C is:

$$\text{KCl conductivity at } 24.0°C = 651 \times [1 + 0.021 \times (24.0 - 20.0)]$$
$$= 706\ \mu S$$

$$\text{cell constant} = \frac{706\ \mathit{m}S/cm}{715\ \mathit{m}S}$$
$$= 0.987\ /cm$$

8.2 Brix

Dilution factor:

mass of molasses in stock solution	=	20.19 g
mass of total solution	=	100.02 g
dilution factor	=	4.95

Brix at 20.0°C:

Brix of the solution at 20.0°C	=	17.9°Bx
multiply by the dilution factor	=	17.9°Bx × 4.95
Brix of the molasses	=	88.6°Bx

8.3 Sample conductivity ash

mass of sample used	=	5.02 g
conductivity for water at 25.0°C	=	1.17 µS
conductivity for sample at 25.0°C	=	449.22 µS
conductivity	=	(449.22 - 1.17) µS
	=	448.05 µS

$$\text{ash (\%) at } 25.0°C = (16.2 + 0.36 \times \frac{88.6}{100} \times \frac{5.05}{20}) \times 448.05 \times 0.987 \times \frac{5}{5.02/20} \times 10^{-4}$$
$$= 14.342\%$$

$$\text{ash (\%) at } 20.0°C = \frac{14.402\%}{[1 + 0.023 \times (25.0 - 20.0)]}$$

$$= 12.863\%$$

Report as 12.86%

9. References

ICUMSA (1994). Conductivity ash in raw sugar, brown sugar, juice, syrup and molasses. *ICUMSA Methods Book,* Method GS1/3/4/7/8/-13.

SASTA (1985). *Laboratory Manual for South African Sugar Factories.* 3rd Edition: 156 - 157, 311.

SMRI (2002). The determination of conductivity ash in raw sugar, brown sugar, juice, syrup and molasses. *SMRI Test Methods,* TM066.