Method 7.6 – Raw sugar: reducing sugars by the Luff Schoorl method

1. Rationale

The method is applicable to all raw sugars and is used to determine the amount of reducing sugars (mainly fructose and glucose) in the sample.

2. Principle

The sample is dissolved in water and reacted with a known amount of copper (II) in solution (Luff Schoorl reagent). The reducing sugars in solution will reduce the copper (II) to copper (I) in an equimolar ratio. The excess copper (II) is determined by titration with sodium thiosulphate.

3. Definitions

3.1 Reducing sugars

Reducing sugars consist primarily, but not exclusively, of glucose and fructose and are obtained through the hydrolysis of sucrose.

3.2 Invert: an equimolar mixture of glucose and fructose.

4. Apparatus

4.1 Stopwatch

4.2 Fine pumice to prevent over-boiling

4.3 Pipettes: 10, 15 and 25 cm^3

4.4 Reflux condenser with a ground glass joint

4.5 Conical flask: 250 cm^3 with a ground glass joint to fit the condenser

4.6 Burette: 50 cm^3

4.7 Measuring cylinders: 3×25 cm^3

4.8 Light duty balance: readable to 0.01 g

4.9 Analytical balance: readable to 0.0001 g

4.10 Hotplate capable of bringing a solution to the boil within 2½ minutes

4.11 Cooling bath with running water

4.12 Volumetric flasks: 100, 1 000 and 2 000 cm^3

4.13 Beakers: 100, 3×250, 500, 1 000 and 2 000 cm^3

5. Reagents

5.1 Copper sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$)

5.2 Citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$)

5.3 Sodium carbonate (anhydrous)

Sodium carbonate (Na_2CO_3) is a corrosive base and contact with the skin and eyes must be avoided. Wear gloves and safety glasses during use.

5.4 Luff School reagent

Solution A: weigh 34.6 g analytical grade copper sulphate pentahydrate and 230 g citric acid monohydrate and dissolve in 400 cm^3 distilled water in a 1 000 cm^3 beaker.

Solution B: weigh 370.6 g anhydrous sodium carbonate and dissolve in 1 000 cm^3 distilled water in a 2 000 cm^3 beaker.

Carefully add solution B to solution A with constant stirring. Transfer to a 2 000 cm^3 volumetric flask and make to the mark.

5.5 Potassium iodate

Potassium iodate (KIO_3) is an irritant and should only be handled while wearing gloves and safety glasses. Toxic fumes may form above 100°C.

Dry the potassium iodate overnight in a desiccator before use.

5.6 Potassium iodide powder

Potassium iodide (KI) is an irritant and should only be handled while wearing gloves and safety glasses.

5.7 Potassium iodide solution (20%)

This reagent is unstable and must be prepared immediately prior to use.

Weigh 20 g potassium iodide (KI) into a 250 cm^3 beaker, dissolve in distilled water and make to approximately 100 cm^3. This should be sufficient for six titrations.

5.8 Sulphuric acid (1:5)

Sulphuric acid (H_2SO_4) is a corrosive acid and contact with the skin, eyes and through ingestion must be avoided. Wear gloves and safety glasses during use.

Add 1 part of concentrated sulphuric acid carefully to 5 parts of distilled water. (For example: add 10 cm^3 to 50 cm^3 distilled water.) Always add the acid to the water and not the other way around. The dilution is exothermic and the solution will heat. Cool the solution to room temperature before bottling.

5.9 Sulphuric acid (1:9)

Add 1 part of concentrated sulphuric acid (H_2SO_4) carefully to 9 parts of distilled water. (For example: add 5 cm^3 to 45 cm^3 distilled water.) Always add the acid to the water and not the other way around. The dilution is exothermic and the solution will heat. Cool the solution to room temperature before bottling.

5.10 Chloroform

Chloroform ($CHCl_3$) is a flammable and toxic solvent and must be handled with care. Use in a fume cupboard while wearing gloves and safety glasses.

5.11 Sodium thiosulphate (0.1 M)

Boil about 1 100 cm^3 distilled water for 10 minutes to remove dissolved carbon dioxide (CO_2) and cool to room temperature. Weigh 24.82 g analytical grade sodium thiosulphate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) and dissolve in some of the boiled water in a 500 cm^3 beaker. Transfer to a 1 000 cm^3 volumetric flask and add approximately 5 cm^3 chloroform as a preservative. Make to the mark using the boiled water.

5.12 Starch indicator (1%)

Weigh 1 g soluble starch into a 100 cm^3 beaker and add 20 cm^3 distilled water. Boil the solution for 1 minute. Cool, transfer to a 100 cm^3 volumetric flask and make to the mark with distilled water.

6. Procedure

6.1 Standardisation of the sodium thiosulphate solution

Weigh accurately between 0.075 and 0.085 g dried potassium iodate (KIO_3) in the conical flask and record the mass. Add about 25 cm^3 distilled water and swirl to dissolve. Add 2 g potassium iodide (KI) powder and swirl to dissolve. Add 5 cm^3 of the 1:9 sulphuric acid solution and titrate immediately with the 0.1 M sodium thiosulphate until the solution has a pale yellow colour. Add a few drops of the starch solution as indicator. The solution should turn blue. Continue the titration slowly until the solution is just colourless. Repeat the titration and use an average of the duplicate titres.

$$\text{Concentration } Na_2S_2O_3 \text{ (M or mole/litre)} = \frac{\text{mass of potasium iodate (g)} \times 1000}{35.67 \text{ g/mole} \times \text{titre (cm}^3\text{)}}$$

6.2 Sample preparation

Pipette 25 cm^3 of the Luff Schoorl reagent into the conical flask and add a pinch of pumice. Weigh 20.00 ± 0.02 g of well-mixed raw sugar into a 100 cm^3 volumetric flask. Dissolve in distilled water and make to the mark with distilled water. Pipette 25 cm^3 of each sugar solution (sample aliquot) into the Erlenmeyer flask containing the Luff Schoorl solution and pumice. [If the pol of the sugar is 99.00°Z or below, pipette 10 cm^3 of the sugar solution (sample aliquot) and add 15 cm^3 distilled water.]

Fit the flask to the condenser and place on the hotplate. Heat so that the solution reaches boiling point within 2 to 3 minutes and boil for exactly 5 minutes. Cool the solution under running water. Add 15 cm^3 potassium iodide solution and slowly add 25 cm^3 of the 1:5 sulphuric acid solution with gentle swirling. Titrate against the sodium thiosulphate solution. When the solution becomes a pale straw colour, add approximately 5 drops of starch indicator and titrate until the blue colour turns milky white or colourless. Repeat the titration and use an average of the duplicate titres.

Prepare a blank using 25 cm^3 of distilled water in place of the sugar solution and follow the same procedure.

7. Calculations

Subtract the titre of the blank from that of the sample to give the titre volume (cm^3). Depending on the sample aliquot, use either one of the equations shown below to obtain the mass (mg) of reducing sugars in solution that is equivalent to the volume at the particular sodium thiosulphate solution concentration.

For a sample aliquot of 25 cm^3 (pol > 99.00°Z):

$$\text{rs mass (mg)} = a + b\times V + c\times V^2 + d\times 10^{-2}\times V^3 + e\times 10^{-3}\times V^4 + f\times 10^{-4}\times V^5 + g\times 10^{-5}\times (10\times M\times V)^6$$

where V ≡ volume (titre - blank) (cm^3)
M ≡ sodium thiosulphate concentration (M or mole/litre)

and a = -0.7012412
b = 3.639119
c = -0.3670057
d = 9.4511543
e = -1.179675
f = 7.03517
g = -1.578679

$$\text{reducing sugars \% sugar} = \frac{\text{rs mass (mg)}}{\text{sample mass (g)}\times 1000}\times\frac{100\ cm^3}{25\ cm^3}\times 100$$

For a sample aliquot of 10 cm^3 (pol ≤ 99.00°Z):

$$\text{rs mass (mg)} = a + b\times V + c\times V^2 + d\times 10^{-2}\times V^3 + e\times 10^{-3}\times V^4 + f\times 10^{-4}\times V^5 + g\times 10^{-5}\times (10\times M\times V)^6$$

where V ≡ volume (titre - blank) (cm^3)
M ≡ sodium thiosulphate concentration (M or mole/litre)

and a = -0.164413
b = 3.346176
c = -0.2275891
d = 6.568474
e = -9.122059
f = 5.857626
g = -1.38145

$$\text{reducing sugars \% sugar} = \frac{\text{rs mass (mg)}}{\text{sample mass (g)}\times 1000}\times\frac{100\ cm^3}{10\ cm^3}\times 100$$

Report as percentage to 2 decimal places.

8. Example

Sodium thiosulphate concentration = 0.100 mole/litre
Sample mass = 20.05 g
Titre of blank = 17.3 cm^3

Titre of solution	=	14.0 cm^3
Volume	=	3.3 cm^3

For a 25 cm^3 aliquot:

mass of reducing sugars	=	8.9 mg
reducing sugars in sample	=	$\frac{8.9\ \text{mg}}{20.05 \times 1000} \times \frac{100\ \text{cm}^3}{25\ \text{cm}^3} \times 100$
	=	0.18%

9. Precision

The tolerance associated with the analysis is ± 0.03 unit.

10. References

ICUMSA (1994). Reducing sugars in cane raw sugar by the Luff Schoorl procedure. *ICUMSA Methods Book,* Method GS1-5.

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