DRAFT TANZANIA STANDARD

Determination of Reducing Sugars in Purified Sugar

TANZANIA BUREAU OF STANDARDS

0. Foreword

This draft Tanzania standard prescribes the method for determination of Reducing Sugars in Purified Sugar.

In the preparation of this draft Tanzania standard assistance was derived from ICUMSA Method GS2/3/9-5 (2011) -The Determination of Reducing Sugars in Purified Sugar by the Knight and Allen EDTA Method - Official (Reference) Method

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with TZS 4.

1.Scope

This draft Tanzania standard prescribes the method for determination of Reducing Sugars in Purified Sugar.

2. Field of Application

This method is suitable for the determination of low reducing sugar contents, e.g. in white sugars including specialty white sugars and plantation white sugars up to 0.05%. Such low levels cannot satisfactorily be determined by titration against Fehling's solution. This method is designated the ICUMSA Official (Reference) method, It is the method that ICUMSA recommends for the Determination of Reducing Sugars in White Sugar and Plantation White Sugar in preference to GS2/9-6 Method.

3. Principle

A solution of the sugar is heated in a boiling water bath with an alkaline copper reagent. The cupric ions are reduced to insoluble cuprous oxide by the reducing sugars present. After cooling the residual cupric ions are titrated with EDTA using murexide as an indicator.

4. Reagents

WARNING AND SAFETY PRECAUTIONS

USERS OF THIS METHOD ARE ADVISED TO CONSULT THEIR NATIONAL HEALTH AND SAFETY LEGISLATION AND CHEMICAL SUPPLIERS BEFORE HANDLING THESE REAGENTS. IN PARTICULAR TAKE PRECAUTIONS NOT TO IN-HALE FINE METHYLENE BLUE POWDER DURING GRINDING IN STEP 3.3.

Use analytical grade reagents unless otherwise specified

4.1 Alkaline copper reagent**.** Dissolve 25g of sodium carbonate and 25g of potassium sodium tartrate (Rochelle salt) in about 600 mL of water which contains 40.0 mL of 1.0 mol/L sodium hydroxide in a 1 L flask. Dissolve 6.000 g of cupric sulphate ($CuSO_4 \cdot 5H_2O$) in about 100 mL of water and add quantitatively to the alkaline tartrate solution. Dilute the mixture to 1 L and mix thoroughly.

4.2 EDTA solution, 0.005 mol/L. Prepare a solution containing 1.860 g per litre of the disodium salt of ethylenediaminetetra acetic acid (EDTA) as required and discard after use. Alternatively, dilute 50 mL of 0.01 mol/L EDTA solution (commercially available) to 100 mL as required.

4.3 Murexide indicator. Prepare by grinding together 0.5 g of murexide (ammonium purpurate) which is in powder form, with 0.15 g of powdered methylene blue and 40 g of sodium chloride.

The indicator is best stored in a desiccator over silica gel because humid conditions tend to cause caking. A solution of the indicator is not stable.

NOTE- Both murexide and methylene blue are only available as general-purpose reagent or indicator grades.

4.4 Sucrose - containing less than 0.002% reducing sugars (referred to as 'low-invert').

5. Apparatus

5.1 Analytical balance readable to 2 mg.

5.2 Test tubes - 150 mm x 20 mm.

5.3 White porcelain dish for titration, allows for easier end point detection.

5.4 Water baths- one maintained at the boiling point, preferably with some means of holding the tubes, i.e. holes in the lid, the other to contain tap water at room temperature or below.

5.5 Burettes and pipettes.

6. Procedure

6.1 Weigh accurately 5.0 g of sugar into a test tube and dissolve in 5 mL of distilled water by shaking without warming. Add exactly 5 mL of the alkaline copper solution (3.1). Thoroughly mix the tube and immerse in a boiling water bath for 5 min, then cool immediately in a cold bath.

6.2 Transfer the tube contents and washings to a white porcelain dish, add approximately 0.1 g of the indicator by means of a small spatula or flattened glass rod and mix.

NOTE - If the tube and contents are left to stand before titration, the precipitated cuprous oxide will gradually redissolve

6.3 Titrate the solution with the EDTA solution (3.2) whilst stirring with a magnetic stirrer or a glass rod. The EDTA should be added from the burette to the porcelain dish continuously through the titration procedure. Maintain the stirring of the solution at all times. As the end point is approached, the rate of addition of EDTA can be slowed to 2-3 drops per second but should never be stopped, until the end point is reached. The reason for this is that the colour formed can disappear due to oxidation, meaning that if the titration is stopped an incorrect estimation of the end point may be made, after restarting the titration.

The colour change during the titration is gradual. The solution starts green, will tum to grey, and finally to purple. These changes are gradual, but the end point can be considered to have been reached when the first full purple colour (i.e., when the body of the whole solution is purple) has been reached. On stopping the titration, there should be no concern if the colour formed starts to disappear. This is due to oxidation, and no more EDTA should be added.

Every sample should be analyzed in duplicate to ensure accurate estimations of reducing sugars. Record the mean of the two titres, T mL.

Titration Volume T mL EDTA solution (3.2)	% Reducing Sugars
3.2-3.8	0.050
3.9-4.4	0.048
4.5-5	0.046
5.1-5.7	0.044
5.8-6.3	0.042
6.4-7	0.040
7.1-7.6	0.038
7.7-8.2	0.036
8.3-8.9	0.034
9-9.5	0.032
9.6-10.1	0.030
10.2-10.8	0.028
10.9-11.4	0.026
11.5-12	0.024
12.1-12.7	0.022
12.8-13.3	0.020
13.4-14	0.018
14.1-14.6	0.016
14.7-15.2	0.014
15.3-15.9	0.012
16-16.5	0.010
16.6-17.l	0.008
17.2-17.8	0.006
17.9-18.4	0.004
18.5-19.1	0.002

NOTE- Some plantation white sugars may contain in excess of 0.05% reducing sugars. In these cases, take 2 g of sample, add 3 g of sucrose (3.4) and proceed as in section 5.1. When calculating in section 6.1, multiply the result by 5/2.

7. Expression of Results

7.1 Calculation**.**

The reducing sugars content is obtained either from the Table above or preferably from a calibration graph derived by adding known amounts of invert sugar to low invert sucrose (3.4) and carrying out the above procedure.

Prepare a 10g/L invert sugar solution as detailed in ICUMSA Method GS1/3/7-3. Dilute to make a 0.5g/L invert sugar solution and prepare standards of 0.05, 0.04, 0.03, 0.02, 0.01 and 0.000% invert by adding appropriate amounts of this invert sugar solution to clean dry test tubes and making up to 5 mL. Add 5g of low invert sucrose and carry out the above procedure.

The graphical method shows the relationship to be linear up to 0.05g reducing sugars/100g sucrose.

7.2 Precision

For white sugars containing between 0.009% and 0.04% reducing sugars, the absolute difference between two results obtained under repeatability conditions should not be greater than 0.004%. The absolute difference between two results obtained under reproducibility conditions should be not greater than 0.007%.