DRAFT TANZANIA STANDARD

Determination of Reducing Sugars in White Sugar and Plantation White Sugar

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TANZANIA BUREAU OF STANDARDS

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0. Foreword

This draft Tanzania standard prescribes the method for determination of Reducing Sugars in White Sugar and Plantation White Sugar

In the preparation of this draft Tanzania standard assistance was derived from Method GS2/9-6 (2011) The Determination of Reducing Sugars in White Sugar and Plantation White Sugar by the Modified Ofner Titrimetric Method – Official.

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 White Sugar and Plantation White Sugar

2. Field of Application

The method is applicable to white sugar and plantation white sugar containing up to 0.089% reducing sugars.

If a choice is available to the user for the Determination of Reducing Sugars in White Sugar and Plantation White Sugar, then ICUMSA recommends that Method GS2/3/9-5 is used preferentially.

The method measures the reducing power of solutions of white sugar containing reducing substances eg. invert sugar in a weak alkaline solution of a Cu^{2+} complex with tartrate.

3. Definitions

3.1 Reducing sugars- are mono- and oligosaccharides containing a free aldehydic or ketonic group which show a reducing effect on certain oxidising agents.

3.2 Invert sugar is an equimolar mixture of glucose and fructose.

3.3 Reducing substances - are the sum of reducing sugars and other substances in sugar products defined by their reducing power on reagents which are used for the determination of reducing sugars. Like reducing sugars their amount is in most cases expressed as the equivalent amount of invert sugar, that is the amount of invert sugar which shows the same reducing power under the conditions of the reaction.

4. Principle

4.1. The complex formed between Cu^{2+} ions and potassium sodium tartrate is reduced by reducing sugars to univalent Cu^{+} which is precipitated as Cu_2O. The precipitated Cu_2O is then determined by iodometric titration. The Cu_2O is oxidised by

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an excess of iodine in acid solution to Cu^{2+} and the excess is back-titrated with sodium thiosulphate.

The reaction between the reducing sugars and the Cu^{2+} complex is not stoichiometric. The amount of Cu_2O formed depends upon the prescribed reaction conditions which therefore must be strictly followed.

It has been determined that 1 mL 0.01615 mol/L iodine solution is equivalent to 1 mg of reducing sugars, once the correction for the reducing effect of sucrose has been taken into account.

4.2. The modification of the original Ofner method, by A Emmerich, consists of adopting the following characteristics of the Berlin Institute Method:

4.2.1. the amount of copper in the Ofner reagent is in-creased by 40% to extend the measuring range of the method from less than 20 mg to 25-30 mg.

4.2.2. a blank value is introduced which takes account of the influence of impurities in the reagents.

4.2.3. a cold value is introduced to allow for reducing substances in the sample, other than reducing sugars, forming Cup and reacting with iodine at room temperature.

4.3. One of the major advantages of the Ofner method compared with the Berlin Institute method is that the sucrose correction, which allows for the reducing power of sucrose, amounts to only 1 mg for every 10 g of sucrose. This is half of the correction used in the Berlin Institute method and it is much more constant and reliable.

5 Reagents and Materials

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.

Use only distilled water or water of similar quality. All reagents should be of analytical grade or better unless stated.

5.1 Activated Carbon - powdered.

5.2 Small pumice pieces.

5.3 Disodium hydrogen phosphate dodecahydrate $Na_2HPO_4 \cdot 12H_20$.

5.4 Glacial acetic acid, ρ_{20}, = 1.05 g/mL.

5.5 Acetic acid solution, approx. 5 mol/L.

5.6 Potassium sodium tartrate tetrahydrate (Rochelle or Seignette salt), $KOOC\text{-}CH(OH)\text{-}CH(OH)\text{-}COONa \cdot 4H_2O$.

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5.7 Copper sulphate pentahydrate, $CuSO_4 \cdot 5H_2O$.

5.8 Sodium carbonate, anhydrous, Na_2CO_3,

5.9 Soluble starch.

5.10 Hydrochloric acid, approx. 1 mol/L.

5.11 Hydrochloric acid, approx. 2 mol/L.

5.12 **Ofner solution, modified**. Weigh out 7.0 g copper sulphate pentahydrate (5.7), 10.0 g sodium carbonate (5.8), 300 g potassium sodium tartrate (5.6) and 50 g disodium hydrogen phosphate (5.3) in a 1000 mL flask. Dissolve in approx. 900 mL water (heating slightly to dissolve if necessary). Heat the solution for 2 hours in a boiling water bath. Cool down to room temperature and make up to the mark. Add approx. 10 g activated carbon (5.1) and stir for 5-10 min. Filter the solution (6.11).

5.13 **Potassium iodate solution 0.01667 mol/L**. Dry the potassium iodate for 3 hours at 100 °C before use. Weigh out 3.5667 g potassium iodate, KIO_3. Transfer to a 1000 mL volumetric flask, dissolve in water and make up to the mark.

5.14 Starch solution (indicator for iodine). Dissolve 1 g of soluble starch in 100 mL saturated sodium chloride solution. Bring the solution to the boil for a few minutes.

5.15 Potassium iodide, KI.

5.16 Sodium thiosulphate, $Na_2S_2O_3 \cdot 5H_2O$.

5.17 Iodine, I.

5.18 Sodium thiosulphate solution, 0.1 mol/L. Weigh out 24.818 g of sodium thiosulphate (5.16). Transfer to a 1000 mL volumetric flask and dissolve in 400 mL of distilled water. Make up to the mark with water. Alternatively, use ampoules, e.g, Merck Art 1.09950.

5.19 Sodium thiosulphate solution, 0.0333 mol/L. Dilute a 0.1 mol/L sodium thiosulphate solution threefold with water and standardize with potassium iodate. Dissolve 2 g of potassium iodide in 10 mL water. Add 5 mL of approx. 2 mol/L hydrochloric acid (5.11) and 10.0 mL of 0.01667 mol/L potassium iodate solution (5.13). Cover the Erlenmeyer flask with a watch glass, shake gently and leave the solution in the dark for approx. 30 min. Titrate the iodine formed with the sodium thiosulphate solution (5.19) to complete decolorization adding 1 mL of starch indicator (5.14) immediately before the endpoint.

Calculate the factor f_{Th}, of the thiosulphate solution

$$f_{Th} = \frac{30.96}{V_{Th}}$$

V_{Th} =Volume (mL) of sodium thiosulphate solution titrated.

NOTE -f_{Th}, corrects the used iodine solution to the experimentally determined value of 0.01615 mol/L, for which 1 mL corresponds to 1 mg reducing sugars.

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5.20 Iodine solution, 0.05 mol/L. Dissolve 53 g of potassium iodide (5.15) in 50 mL of distilled water in a 1000 mL volumetric flask. Transfer 12.690 g of iodine (5.17) into this flask, dissolve and make up to the mark with water. Keep this solution protected from light. Alternatively, use an ampoule for preparation, e.g., Merck Art. 1.09910.

5.21 Iodine solution 0.01667 mol/L. Dilute the 0.05 mol/L iodine solution (5.20) threefold with water and standardize with the 0.0333 mol/L sodium thiosulphate solution (5.19). Pipette 25.0 mL of the 0.01667 mol/L iodine solution into a 300 mL Erlenmeyer flask. Add 5 mL of 5 mol/L acetic acid (5.5) and, after gently shaking the mixture, back titrate with the 0.0333 mol/L sodium thiosulphate solution (5.19). Add 1 mL of starch indicator (5.14) just before the endpoint is reached. Calculate the factor f_1, of the iodine solution:

$$f_1 = \frac{V_{Th} - f_{Th}}{25}$$

V_{Th} = Volume (mL) of sodium thiosulphate solution titrated

f_{Th} = correction factor for the sodium thiosulphate solution

6 Apparatus

6.1 Analytical balance - readable to 0.1 mg.

6.2 Precision balance readable to 0.1 g.

6.3 Burettes - capacity 50 mL.

6.4 Erlenmeyer flasks - capacity 300 mL.

6.5 Volumetric flasks - 1000 mL and 200 mL.

6.6 Pipettes - capacities 1 mL, 15 mL and 50 mL.

6.7 Watch glasses to cover Erlenmeyer flasks.

6.8 Bunsen burner, tripod stand and wire gauze.

6.9 Boiling water bath.

6.10 Water bath with cold running water.

6.11 Filter paper.

7 Procedure

7.1 Preparation of the sample. The solution prepared for the determination should contain not more than 25 mg invert sugar in 50 mL. This requires that 40 g of white sugar be made up with water to 200 mL.

7.2 "Hot Value". Mix 50.0 mL of the prepared solution (7.1) in an Erlenmeyer flask with 50.0 mL of the Ofner solution (5.12). Add some pumice pieces (5.2) to the mixture.

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(a) Bring the mixture to boil within 4 to 5 minutes using the Bunsen burner, tripod stand and wire gauze. Boil for exactly 5 min. Note the start of boiling is once numerous steam bubbles break over the whole surface. Cool the mixture down in a water bath with cold running water. After approx. 10 min. the mixture should have reached room temperature.

(b) Add 1 mL concentrated acetic acid (5.4). Add iodine solution (5.21) until the colour of the mixture turns a typical iodine colour. This procedure dissolves the formed Cu_2O with an excess of iodine. Record this volume as V_1,

(c) Add 15 mL of the 1 mol/L hydrochloric acid (5.10) by pouring it down the inner side of the flask so that residual droplets are washed down into the solution. Cover the flask with a watch glass and move it gently for 2 min. until the precipitate of Cu_2O is completely dissolved.

(d) Titrate the sample with 0.0333 mol/L sodium thiosulphate (5.19). Add 1 mL of starch solution immediately before the endpoint is reached. Record this volume as v_2,

(e) Repeat steps (a- d) with another prepared solution mixed with Ofner solution and record the average of the two replicates V_1, and V_2, for iodine and thiosulphate respectively.

7.3 "**Cold value".** Mix 50.0 mL of the prepared sample (7.1) with 50.0 mL of the Ofner solution (5.12).

Leave the mixture at room temperature for 10 min.

Repeat step (b-d) in 7.2. Record values V_3, and V_4, for iodine and thiosulphate respectively.

7.4 "Blank value". Mix 50.0 mL of water with 50.0 mL of the Ofner solution (5.12). Repeat the steps (a-d) in 7.2. Record the values V_5, and V_6, for iodine and thiosulphate respectively.

NOTE- It is essential that the time between addition of iodine solution and beginning of the back titration is equal for the "Hot value" and the "Cold value".

8 Expression of Results

8.1 Calculation of the results

Added amount of iodine for "Hot value" = V_1

Added amount of thiosulphate for "Hot value" = V_2

Added amount of iodine for "Cold value" = V_3

Added amount of thiosulphate for "Cold value" = V_4

Added amount of iodine for "Blank value" = V_5.

Added amount of thiosulphate for "Blank value" = V_6

Corrected consumption of 0.01667 mol/L iodine solution

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Calculated "Hot value", A = $(V_1 \cdot f_1) – (V_2 \cdot f_{Th})$

Calculated "Cold value", B = $(V_3 \cdot f_1) – (V_4 \cdot f_{Th})$

Calculated "Blank value", C = $(V_5 \cdot f_1) – (V_6 . f_{Th})$

where:

f_1 = the factor of the iodine solution, calculated in 5.21,

and Sucrose correction D, is 0.1 mL iodine solution/g of sucrose in the reaction mixture.

$$\text{Invert sugar, mg/kg} = \frac{(A - B - C - D) \cdot 1000}{s}$$

Where:

s = the amount of sample in 50 mL of prepared solution (7.1).

8.2 Example Calculation.

40 g white sugar is weighed out and diluted to 200 mL. 50.0 mL of this solution contains 10 g sucrose.

Amount of iodine solution added to hot value is 20.00 mL.

Amount of sodium thiosulphate consumed is 18.80 mL.

Amount of iodine solution added to cold value is 20.00 mL.

Amount of sodium thiosulphate consumed is 19.90 mL.

Amount of iodine added to the blank value is 20.00 mL.

Amount of sodium thiosulphate consumed is 20.00 mL.

f_{Th} is calculated to be 1.029.

f_1, is calculated to be 1.031.

A = (20.00 - 1.031) - (18.80 -1.029) = 1.27

B = (20.00 -1.031)- (19.90 -1.029) = 0.14

C = (20.00 -1.031) - (20.00 - 1.029) = 0.04

D = 10.0 - 0.1 = 1.00

$$\text{Reducing sugar mg/kg} = \frac{(1.27 - 0.14 - 0.04 - 1.00) \cdot 1000)}{} = 9.0 \text{ mg/kg}$$

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8.3 Precision

For white sugars and plantation white sugars containing between 0.007% and 0.089% reducing sugars, the absolute difference between two results obtained under repeatability conditions should not be greater than 0.006%. The absolute difference between two results obtained under reproducibility conditions should be not greater than 0.011 %.

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