Sulphur Dioxide

1. PURPOSE

This document describes the procedure to be followed for determination of sulphite in sugar.

2. SCOPE

This procedure applies to sugar and sugar syrup.

3. RESPONSIBILITIES

All Analysts carrying out the analysis.

4. REFERENCE

4.1 This document is prepared from method GS 2/1/7 – 33 (2009) – ICUMSA Method Book.

4.2 Vogel's Text Book of Quantitative Chemical Analysis

5. REAGENTS

5.1 Rosaniline hydrochloric solution (saturated) : Suspend 1g of rosaniline hydrochloride in 100 ml of distilled water, heat to 50 °C and cool with shaking. After standing for 48 hr, filter the solution.

5.2 Decolourised rosaniline solution : Transfer 4 ml of saturated rosaniline hydrochloride solution to a 100 ml volumetric flask. After addition of concentrated hydrochloric acid (6 ml), make the mixture up to the mark. Decolourisation takes places in a short time but allow the solution to stand for at least 1 hr. before use.

5.3 Formaldehyde solution (approx. 0.2g/100 ml) : Dilute 5 ml of analytical reagent grade formaldehyde solution ($\rho_{20} \approx$ 1.070 - 1.080) to 1000 ml.

5.4 Pure Sucrose solution : Dissolve 100 g of sulphite - free sucrose in water and make up to 1000 ml.

5.5 Sodium hydroxide solution, 0.1 mol/ L.

5.6 Iodine solution, 0.05 mol/l (0.1N) : Prepare from ampoules of Qualigen CVS orTake 20g potassium iodide and dissolve in 30 to 40 ml distilled water then add 12.7g resublimed iodine. Insert glass stopper into the flask and shake in the cold until all iodine has dissolved. Allow solution to aquire room temp and make upto 1000 ml.

Standardization of Iodine Solution (0.05 mol/L) 0.1 N

Take 25 ml of the iodine solution to a 250 ml conical flask, dilute to 100 ml and add the standard thiosulphate solution from burette until the solution has a pale – yellow colour. Add 2 ml of starch solution and continue the addition of thiosulphate solution slowly until the solution is just colourless. Calculate normality of Iodine.

5.7 Concentrated hydrochloric acid, $\rho_{20} \approx$ 1.18 g/ml.

5.8 Hydrochloric acid solution, approx. 1 mol/L

5.9 Starch indicator: Weigh 1g starch and make slurry in small quantity of water and add this slurry in 100 ml boiled water.After cooling use as a starch indicator.

5.10 Sodium thiosulphate solution, 0.1 mol/L. : Dissolve 6.2042 g of analytical reagent grade sodium thiosulphate pentahydrate in 50 ml of distilled water in 250 ml volumetric flask and then make up to the mark.

Standardisation of Sodium thiosulphate (0.1 mol/lit) with standard potassium dichromate.

Standard $K_2Cr_2O_7$ 0.1 mol/L. –

Dry standard $K_2Cr_2O_7$ (Secondary standard reference material, AR grade) in a oven at 120^0C for 2 hrs. Cool in desicator. Weigh exactly 0.4903 gm $K_2Cr_2O_7$, dissolve in distilled water and dilute to 100 ml with distilled water in a class A volumetric flask.

Standardization:-

Take 100 ml cold, recently boiled distilled water in a 500 ml conical flask. Add 3 gm of potassium iodide & 2 gm of sodium hydrogen carbonate and shake until salts dissolve. Add 6 ml of conc. HCl slowly whilst gently rotating the flask in order to mix the liquids. Add 25 ml standard 0.1 N $K_2Cr_2O_7$ mix the solutions well, and wash the sides of the flask with a little boiled water. Cover it with a watch glass and allow to stand in the dark for 5 minutes in order to complete the reaction. Rinse the watch glass and dilute the solution with 300 ml cold, boiled out water. Titrate the liberated iodine with the sodium thiosulphate solution from burette using starch as an indicator (2 ml). End point is greenish – blue to light green. Calculate the exact normality of thiosulphate from burette reading.

5.11 Standard sulphite solution

5.11.1 Dissolve approx. 0.2 g of unhydrous sodium sulphite in sucrose solution and make up to 100 ml with pure sucrose solution.

5.11.2 Determine the titre of this solution as follows-Place 25 ml of the 0.05 mol/L iodine solution in conical flask and add 10 ml 1 mol/L hydrochloric acid solution followed by approximately 100 ml of distilled water. Pipette 25 ml of standard sulphite solution in to this flask while swirling the flask. Then titrate the excess iodine with the 0.1-mol/L sodium thiosulphate solution until the contents of the flask are a pale straw colour. Then add the 3-4 drops starch indicator to the flask and continue the titration until the blue colour disappears. Record the titer 't'.

5.12 Dilute Standard sulphite solution

5.12.1 Dilute 5 ml of standard sulphite solution to exactly 100 ml with pure sucrose solution.

5.12.2 The exact value of the sulphite content, C, is calculated as follows from the titre, t

$$C = (25 - t) \times 3.203 \times 2 \quad \mu g\ SO_2/ml$$

6. APPARATUS

6.1 Spectrophotometer

6.2 Analytical Balance

6.3 Volumetric flasks- 100 ml class 'A'

6.4 Pipettes –10ml class 'A'

6.5 Pipettes – 2,10 & 25 ml

6.6 Burette

6.7 Test tubes

7. PROCEDURE

7.1 Colour development

7.1.1 Dissolve 10-40 g of a sample of sugar in distilled water in a 100 ml volumetric flask.

For levels 0-5 mg SO2/kg use 40 g of sample

For levels 5-15 mg SO2/kg use 20 g of sample

For levels 15-30 mg SO2/kg use 10 g of sample

7.1.2 After addition of 4 ml 0.1 mol/L sodium hydroxide solution make the contents of the flask up to the mark and mix.

7.1.3 Transfer a 10 ml aliquot to each two clean and dry test tubes.

7.1.4 Add 2 ml of decolourised rosaniline solution and 2 ml of formaldehyde solution and allow the test tubes to stand at room temperature for 30 min.

7.1.5 Measure the absorbance in a 1 cm cell on a Spectrophotometer at 560 nm. For reference add 2 ml distilled water instead of 2 ml decolorized rosaniline Solution in 10 ml diluted sample.

7.2 Standard Curve

7.2.1 Pipette aliquots of the dilute standard sulphite solution 0,0.4,1,2,3,4,5, and 6 ml in to a series of 100 ml volumetric flasks (corresponding to 0, 2, 5, 10, 15, 20, 25 and 30 μg SO_2 .)

7.2.2 To each flask add 4 ml of 0.1 mol/L sodium hydroxide and make the contents up to the mark with 10% pure sucrose solution and mix.

7.2.3 From each flask transfer a 10 ml aliquot to a clean and dry test tube.

7.2.4 Add 2 ml of decolourised rosaniline solution and 2 ml of formaldehyde solution and allow the test tubes to stand at room temperature for 30 min.

7.2.5 Measure the absorbance in a 1 cm cell on a Spectrophotometer at 560 nm using distilled water as a reference and plot the results on graph.

7.2.6 The amount of SO_2 in each test tube is:

$$\frac{c \times n}{10} \ \mu g\ SO_2$$

Where n is the number of ml of dilute sulphite added to each 100 ml flask and C is concentration from 5.12.2 above.

8. Calculation

Calculate the concentration of sulphite by reference to the standard curve and express the results as mg SO_2 / kg in sugar as follows :

$$\mu g \text{ of } SO_2 \text{ from graph} = \frac{\text{Crrected absorbance - intercept}}{\text{Slope}}$$

Corrected absorbance = Absorbance for sample – Sample blank

$$SO_2 \text{ Content in mg/ kg} = \frac{(\mu g \text{ of } SO_2 \text{ from graph }) \times 10}{\text{Wt. of Sample in g}}$$