Method 8.10 – Refined sugar: sulphite (SO_2) by the Rosaniline method

1. Rationale

The method is applicable only to refined sugar and is based on the colourimetric determination of the amount of sulfite in the sample, measured as SO_2.

2. Principle

The sugar is dissolved and reacted with formaldehyde and a rosaniline solution. The colour of the sulphite/rosaniline complex is measured spectrophotometrically at a wavelength of 560 nm and compared to a standard graph.

3. Apparatus

3.1 **Spectrophotometer** operating at 560 nm

3.2 **Light duty balance** readable to 0.01 g

3.3 **Volumetric flasks**: 100, 200 and 1 000 cm^3

3.4 **Pipettes**: 1, 2, 5, 10 and 50 cm^3

3.5 **Test tubes** approximately 160 mm long and 15 mm ϕ

3.6 **Conical flask**: 500 cm^3

3.7 **Measuring cylinder**: 100 cm^3

4. Reagents

4.1 **Rosaniline-hydrochloric acid solution** (saturated)

Rosaniline hydrocloride ($C_{19}H_{18}ClN_3$) is irritating to the eyes, skin and respiratory tract. Gloves and safety glasses must be worn when preparing the solution.

Suspend 1 g of rosaniline hydrochloride in 100 cm^3 of distilled water, heat to approximately 50°C and cool while shaking to dissolve. After standing for 48 hours, filter the solution and store in a dark coloured bottle.

4.2 **Hydrocloric acid** (concentrated, 32%)

Hydrochloric acid (HCl) is a corrosive acid and should only be handled in a fume cupboard while wearing gloves and safety glasses.

4.3 Decolourised/bleached rosaniline solution

Transfer 4 cm^3 of standard rosaniline hydrochloride solution to a 100 cm^3 volumetric flask. Add 6 cm^3 concentrated hydrochloric acid and make to the mark. Decolourisation takes place in a short time but allow the solution to stand for at least 1 hour before use.

4.4 Formaldehyde solution (0.2 %)

Formaldehyde is a corrosive liquid. Work in a fume cupboard while wearing gloves and safety glasses.

Dilute 5 cm^3 of formaldehyde (assay 35-37%) to 1 000 cm^3 in a volumetric flask with distilled water.

4.5 Sucrose solution (10%)

Dissolve 100 g of first-boiling sugar in water and make up to 1 000 cm^3.

4.6 Sodium hydroxide solution (0.1 M)

Sodium hydroxide (NaOH) is a corrosive base and should only be handled with gloves while wearing safety glasses.

Dissolve 4.0 g sodium hydroxide pellets in distilled water. Cool and dilute to 1 000 cm^3 in a volumetric flask.

4.7 Potasium iodide

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

4.8 Iodine

Iodine (I_2) is a corrosive solid and should only be handled with gloves while wearing safety glasses.

4.9 Iodine solution (0.05 M)

Dissolve 70 g of iodate-free potassium iodide (KI) in 100 cm^3 of distilled water in a 1 000 cm^3 volumetric flask. Add 12.69 g of iodine (I_2), shake the flask until all the iodine has dissolved and make to the mark with distilled water.

4.10 Hydrochloric acid solution (1 M)

Dilute 98 cm^3 of concentrated hydrochloric acid to 1 000 cm^3 in a volumetric flask.

4.11 Starch indicator solution (1%)

Weigh 1 g of 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.

4.12 Chloroform

Chloroform (CCl_4) is a flammable solvent and is toxic to humans. Work in a fume cupboard while wearing gloves and safety glasses.

4.13 **Sodium thiosulphate solution** (0.1 M)

Sodium thiosulphate ($Na_2S_2O_3 \cdot 5H_2O$) is mildly irritating. Wear gloves and safety glasses during use.

Boil about 1100 cm^3 distilled water for 10 minutes and cool to room temperature. Weigh 24.82 g sodium thiosulphate pentahydrate and dissolve in some of the boiled water. Make to 1 000 cm^3 in a volumetric flask using the boiled water. Add approximately 5 cm^3 chloroform to the flask as a preservative.

4.14 **Sodium sulphite** (heptahydrate or anhydrous)

Sodium sulphite (Na_2SO_3) is an irritant and must be handled with gloves while wearing safety glasses.

5. Procedure

5.1 Standard graph

Weigh 0.50 g sodium sulphite heptahydrate and dissolve in approximately 60 cm^3 of the 10% sucrose solution. Transfer quantitatively into a volumetric flask (100 cm^3 if sodium sulphite heptahydrate is used or 200 cm^3 if anhydrous sodium sulphite is used) using the 10% sucrose solution and make to the mark.

Pipette 50 cm^3 of the sodium sulphite solution into a 250 cm^3 conical flask. Add 10 cm^3 of the 1 M hydrochloric acid, 100 cm^3 distilled water and a few drops of starch indicator solution. Swirl to mix. Titrate against the standard iodine solution until the first appearance of a blue colour. Calculate the sulphur dioxide (SO_2) content of the sodium sulphite solution from the equation below.

$$SO_2 \text{ (µg in 50 cm}^3\text{)} = 3.203 \times \text{iodine titre}$$

Dilute 5 cm^3 of the prepared sodium sulphite solution with sucrose solution to 100 cm^3 to give a primary solution with a known concentration of approximately 60 µg SO_2 per cm^3. Prepare calibration standards in 100 cm^3 volumetric flasks using the primary standard volumes indicated in Table 1.

$$SO_2 \text{ of primary standard (µ/cm}^3\text{)} = SO_2 \text{ of sodium sulphite solution} \times \frac{5\ cm^3}{100\ cm^3}$$

Table 1: Calibration standards

Volume of primary standard used (cm^3)	0	1	2	3	4	5	6
SO_2 content (mg/kg on sugar)	0	$\frac{y}{10}$	$\frac{2y}{10}$	$\frac{3y}{10}$	$\frac{4y}{10}$	$\frac{5y}{10}$	$\frac{6y}{10}$

y is the exact concentration of the primary standard in µg SO_2/cm^3

Add 4 cm^3 sodium hydroxide solution to each flask and make to the mark with sucrose solution. Pipette 10 cm^3 of each calibration standard into a test tube. Add 2 cm^3 bleached rosaniline and 2 cm^3 of formaldehyde solution and mix well. Stand at room temperature for 30 ± 5 minutes and measure the absorbance at 560 nm against distilled water as reference. Plot a graph of the absorbance against SO_2 (mg/kg on sugar).

5.2 Sample analysis

Weigh 10.0 ± 0.1 g white sugar in a 100 cm^3 volumetric flask and dissolve in approximately 60 cm^3 of distilled water. Add 4 cm^3 of sodium hydroxide solution and make to the mark with distilled water. Include a blank that contains 4 cm^3 of the sodium hydroxide solution diluted to 100 cm^3 with distilled water. Pipette 10 cm^3 of the sugar and the blank solutions into test tubes. Add 2 cm^3 of the rosaniline solution followed by 2 cm^3 of the formaldehyde solution to each and mix well. Stand at room temperature for 30 ± 5 minutes and measure the absorbance in a 10 mm cell at 560 nm against the blank as reference.

If the SO_2 content of the sample is low, repeat the analysis using 20.0 ± 0.1 g sample and divide the answer by 2.

6. Expression of results

6.1 Standard graph

Subtract the absorbance of the blank (standard 1) from the absorbance of the other six solutions. Plot these absorbance values (AU) against the SO_2 concentrations (mg/kg on sugar). This graph should be a straight line passing through the origin.

6.2 Samples

Subtract the absorbance of the sample blank from the absorbance of the sample solution to get the absorbance of the sample. Read the amount of sulphur dioxide in mg/kg from the standard graph as mg SO_2/kg sugar (*i.e.* mg/kg SO_2).

7. Example

7.1 Sodium sulphite solution concentration

Iodine titre	=	17 cm^3
SO_2 (µg in 50 cm^3)	=	3.203 × iodine titre
	=	54 451 µg in 50 cm^3
	=	1 089 µg/cm^3

$$\text{Primary standard solution concentration} = 1089\ \mu g/cm^3 \times \frac{5\ cm^3}{100\ cm^3}$$

$$= 54.45\ \mu g/cm^3$$

Table 2: Calibration standards

Volume (cm3)	SO_2 content (mg/kg on sugar)	Absorbance (AU)	Absorbance - Blank (AU)
0	0.00	0.023	0.000
1	5.45	0.245	0.222
2	10.89	0.506	0.483
3	16.34	0.713	0.690
4	21.78	0.927	0.904
5	27.23	1.142	1.119
6	32.67	1.327	1.304

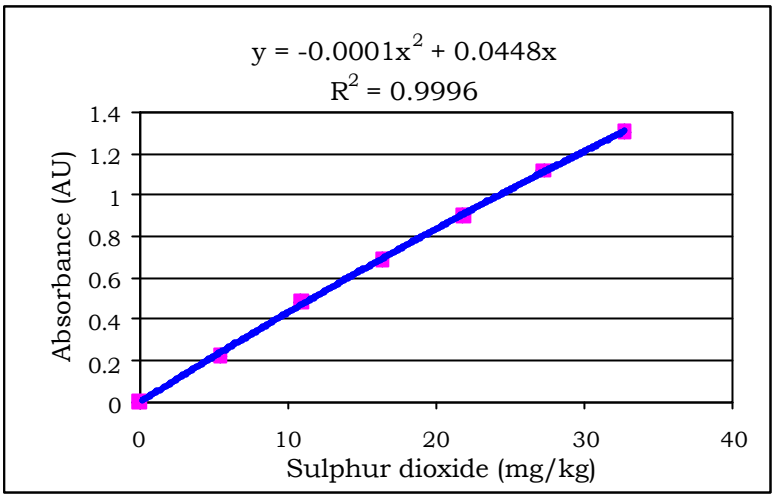

Figure 1: Standard sulphur dioxide graph

7.2 Sample

Absorbance blank	=	0.024 AU
Sample absorbance	=	0.614 AU
Corrected absorbance	=	0.590 AU
SO_2	=	13.78 mg/kg

Report as 14 mg/kg

8. References

ICUMSA (2002). Sulphite in white sugar, cane sugar juices and syrups and very very high pol (VVHP) sugars by the Rosaniline colorimetric method. *ICUMSA Methods Book,* Method GS2/1/7-33.

SASTA (1985). *Laboratory Manual for South African Sugar Factories*. 3rd Edition: 200 - 202, 342.

SMRI (1997). Determination of the sulphite in white sugar by the Rosaniline/hydrochloric colorimetric method. *SMRI Test Methods,* TM061.