



## Method 9.9 – Boiler water: sulphites

### 1. Rationale

The method is applicable to boiler water and determines the amount of sulphite as mg  $\text{SO}_3^{2-}$  per litre in the sample.

### 2. Principle

This method is used to determine small amounts of sulphite in solution. The water sample is mixed with an excess of a standard iodine solution and acidified. The excess iodine is titrated with a standard thiosulphate solution and starch indicator to determine the amount of sulphite in the sample.

### 3. Apparatus

- 3.1 **Burette:** 10 cm<sup>3</sup>
- 3.2 **Pipettes:** 3, 20, 100 cm<sup>3</sup>
- 3.3 **Conical flask:** 250 cm<sup>3</sup>
- 3.4 **Volumetric flasks:** 1 000 cm<sup>3</sup>
- 3.5 **Analytical balance** readable to 0.0001 g

### 4. Reagents

#### 4.1 Potassium iodide

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

#### 4.2 Iodine

*Iodine (I<sub>2</sub>) is a corrosive solid and must be handled with gloves while wearing safety glasses.*

#### 4.3 Iodine solution (0.005 M)

Dissolve 7 g of iodate-free potassium iodide (KI) in 100 cm<sup>3</sup> of distilled water in a 1 000 cm<sup>3</sup> volumetric flask. Add 1.2691 g of iodine (I<sub>2</sub>), shake the flask until all the iodine has dissolved and make to the mark with distilled water.

#### 4.4 Sodium thiosulphate solution (0.01 M)

*Sodium thiosulphate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> · 5H<sub>2</sub>O) is mildly irritating and must be handled with gloves while wearing safety glasses.*

Dissolve 2.4813 g sodium thiosulphate pentahydrate in distilled water and make to 1 000 cm<sup>3</sup> in a volumetric flask.

#### 4.5 Starch indicator solution (1%)

Weigh 1 g of soluble starch into a 100 cm<sup>3</sup> beaker and add 20 cm<sup>3</sup> distilled water. Boil the solution for 1 minute. Cool and make to 100 cm<sup>3</sup> with distilled water.

#### 4.6 Phosphoric acid (concentrated)

*Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) is a corrosive acid and may cause upper respiratory disease. Contact with the skin, eyes and through inhalation should be avoided. Wear safety glasses and gloves during handling.*

### 5. Procedure

Pipette 100 cm<sup>3</sup> boiler water into a 250 cm<sup>3</sup> conical flask and quickly add 3 cm<sup>3</sup> phosphoric acid and 20 cm<sup>3</sup> of the 0.005 M iodine solution. Add a few drops of the starch solution and titrate with the 0.01 M sodium thiosulphate solution until colourless. Record the thiosulphate titre in cm<sup>3</sup>.

### 6. Calculations

$$\text{SO}_3^{2-} \text{ (mg/litre)} = \frac{C_{\text{I}_2} \times \text{MM}_{(\text{SO}_3^{2-})}}{V_s} \times (V_{\text{I}_2} - t)$$

where $C_{\text{I}_2}$	≡	Concentration of I <sub>2</sub> (mole/litre)
$\text{MM}_{(\text{SO}_3^{2-})}$	≡	Molecular mass of SO <sub>3</sub> <sup>2-</sup> (g/mole)
$V_s$	≡	Volume of sample (litre)
$V_{\text{I}_2}$	≡	Aliquot of I <sub>2</sub> (litre)
$t$	≡	sodium thiosulphate titre (litre)

$$\begin{aligned} \text{SO}_3^{2-} \text{ (mg/litre)} &= \frac{0.005 \text{ mole/litre} \times 80 \text{ g/mole}}{0.1 \text{ litre}} \times (0.020 - t) \text{ litre} \\ &= 4000 \text{ mg/litre}^2 \times (0.020 \text{ litre} - t) \end{aligned}$$

Report in mg SO<sub>3</sub><sup>2-</sup> /litre to the nearest unit.

### 7. Example

$$\begin{aligned} \text{I}_2 \text{ titre} &= 7.5 \text{ cm}^3 \\ \text{SO}_3^{2-} &= 4000 \text{ mg/litre}^2 \times (0.020 - 0.0075) \text{ litre} \\ &= 50.00 \text{ mg/litre} \end{aligned}$$

Report as 50 mg/litre

### 8. References

SASTA (1985). *Laboratory Manual for South African Sugar Factories*. 3<sup>rd</sup> Edition: 352 - 353.