

## **TEST PROCEDURE**

### **Determination of**

## **Sulfur Dioxide (Free, Bound and Total) in Wine and Grape Juice**

### **by Aspiration/Oxidation**

#### **1. General Safety Precautions**

**Phosphoric Acid** solution used in this method is corrosive and harmful. Wear gloves and safety glasses when handling.

**Hydrogen Peroxide** is corrosive and a strong oxidant. Wear gloves and safety glasses when handling.

#### **2. Reagent Preparation**

2.1 Hydrogen Peroxide Solution, 0.3% w/v. Dilute 1 mL of 30% w/v Hydrogen Peroxide to 100mL with distilled water. Store in fridge when not in use. Discard after two weeks.

2.2 Ortho Phosphoric Acid solution, 25% v/v. Use solution as provided. Transfer desired quantity to 10 mL Kipps measure/conical flask assembly provided.

2.3 Mixed Indicator Solution. Use solution as provided.

2.4 Sodium Hydroxide solution, 0.01 M. Dilute 10.0 mL of 0.1M Sodium Hydroxide to 100.0 mL with distilled water using pipette and volumetric flask. For extreme accuracy, standardise against 0.01M Hydrochloric Acid solution as described in "**Test Procedure for Standardisation of Sodium Hydroxide**". Please note that Sodium Hydroxide will decompose over time, and its strength should be checked regularly (every 1 to 2 weeks). Keep container tightly sealed when not in use.

2.5 Soap Solution. Mix @ 5mL of dishwashing liquid with 5mL of water.

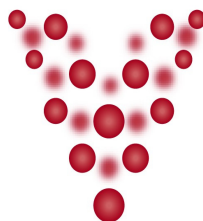
#### **3. Equipment Preparation - Air Flow Standardisation**

This section is important and must be carried out for accurate results.

3.1 The system is to operate under vacuum. The water manometer is set up between the outlet of the pear shaped flask and the vacuum side of the pump. Fill the manometer with water to the black mark.

3.2 After turning on the pump, adjust the flow to 1 L/minute. This is achieved by use of soap bubble flow meter. Attach the tube from the soap bubble meter to the discharge side of the pump. Pour about 2mL of soap solution into the bubble meter chamber, squirt soap solution into the air stream by squeezing the rubber bulb, and with the aid of a stopwatch, measure the time it takes for a soap bubble to move to the 100 mL mark at the top of the chamber. Adjust the knob on the air pump until the time taken for the bubble to travel to the 100 mL mark is 6 seconds. This flow corresponds to a flow rate of 1L/minute.

3.3 When the correct flow rate has been established, mark the water level of the small diameter tube of the Manometer. The bubble meter may now be disconnected, and the markers on the water manometer tube used to set the correct airflow. The airflow should be checked periodically with the bubble meter.



#### 4. Determination of Free Sulfur Dioxide

4.1 To the empty 50 mL pear shape flask, add 0.3% peroxide solution to just above the mark indicated on the side. Use dropper tubes to add 3 to 4 drops of mixed indicator solution, and 1 to 2 drops of dilute Phosphoric Acid (dilute the 25% by approx 100 to 1) solution to the contents of the pear shaped flask. The solution should be a purple colour. Carefully add dropwise 0.01 M NaOH solution until the solution turns olive green. Connect the pear shaped flask to the apparatus. It is not necessary to record the volume of 0.01 M NaOH added.

4.2 To the empty 100 mL round bottom flask, add 20.0 mL of sample with a 20.0 mL pipette followed by 10 mL (Kipps bottle) of Phosphoric Acid solution. Quickly connect the flask to the aspiration assembly.

4.3 Turn on pump and aspirate sample at 1 L/minute for 15 minutes. Note that during this aspiration, it is not necessary to have cooling water flowing through the condenser.

4.4 After 15 minutes, stop the aspiration, carefully disconnect the pear shape flask, and titrate the contents against 0.01 M NaOH until an olive green endpoint is achieved. Record the titre value (A). If an analysis of Bound SO<sub>2</sub> is required, do not discard this solution.

#### 5. Determination of Bound and Total Sulfur Dioxide

5.1 After completing the titration in Step 4.4, reconnect the pear shape flask. Turn on water to the condenser and commence aspirating at 1 L/minute. Heat contents with bunsen burner and boil contents **gently** for 15 minutes.

5.2 After 15 minutes, turn off burner and stop aspirating. Disconnect pear shape flask, and titrate with 0.01 M NaOH as in Step 4.4. Record the titre value (B).

5.3 If Total Sulfur Dioxide only is required, proceed as in Steps 4.1 to 4.4, except that the sample is boiled with the cooling water on. Record the titre value as (C).

#### 6. Calculations

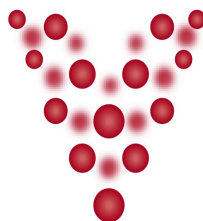
$$6.1 \quad \text{Free SO}_2 \text{ (mg/L)} \quad = \quad \frac{(A) \times M \times 32.06 \times 1000}{V}$$

$$6.2 \quad \text{Bound SO}_2 \text{ (mg/L)} \quad = \quad \frac{(B) \times M \times 32.06 \times 1000}{V}$$

$$6.3 \quad \text{Total SO}_2 \text{ (mg/L)} \quad = \quad \text{Add 6.1 and 6.2 above, **or**}$$

$$= \quad \frac{(C) \times M \times 32.06 \times 1000}{V}$$

Where;        M        =        Molarity of NaOH Solution  
                  V        =        Volume of Sample



## VINTESSENTIAL LABORATORIES - CALCULATION SHEET

### Determination of Free and Bound SO<sub>2</sub> in Grape Juice and Wine

Test Report No: \_\_\_\_\_

Date: \_\_\_\_\_

Molarity of NaOH: \_\_\_\_\_ mol/L

#### Free SO<sub>2</sub>

Sample	Sample Vol. (ml)	Init. Titre Vol. (ml)	Final Titre Vol. (ml)	Total Titre (ml)	Free SO <sub>2</sub> (mg/L)

#### Bound SO<sub>2</sub>

Sample	Sample Vol. (ml)	Init. Titre Vol. (ml)	Final Titre Vol. (ml)	Total Titre (ml)	Bound SO <sub>2</sub> (mg/L)

#### **Calculations:**

$$\text{SO}_2 \text{ Free or Bound (mg/L)} = \frac{\text{Titre} \times M \times 32.06 \times 1000}{V}$$

Where: V = Molarity of NaOH Solution  
          V = Volume of Sample