

TESTS FOR "FREE" AND "BOUND" SULPHUR DIOXIDE

by the RANKINE ASPIRATION METHOD

"Free" Sulphur Dioxide

1. Remove 0.3% Hydrogen peroxide from refrigerator, place 50ml of 0.3% H₂O₂ in a 100ml glass beaker.
2. Place beaker in sink containing warm water. Stir solution with a thermometer to adjust temperature to 20°C.
3. Use a 10ml volumetric pipette to transfer 10ml of 0.3% H₂O₂ to the 2 - necked, pear shaped flask.
4. Add 3 drops of mixed indicator. The solution may then change to a purple colour.
Add 0.01N NaOH to adjust colour to an olive green. Alternatively, the solution may naturally change to a green colour. In this case no addition of 0.01N NaOH is required.
The amount of sodium hydroxide used (if any) for colour adjustment is unimportant and does not have to be recorded. Connect the 2-necked flask to the top of the aspiration apparatus.
5. Adjust temperature of wine sample to 20°C. Using a similar procedure as in steps 1 and 2.
Use a 20ml volumetric pipette to transfer a sample of wine to the 100ml round bottom flask.
6. Also add 10ml of 25% phosphoric acid using a 10ml volumetric pipette to the 100ml round bottom flask.
7. Connect tubing from air pump assembly to the air bleed tube.
8. Turn on the air pump and adjust airflow rate using the plastic valve on the air pump assembly.
The airflow into the 2-neck flask should not be too fast or too slow (theoretically 1L/min).

PLEASE NOTE: The advantage of the air pump assembly is that the volume of air produced by the air pump is exactly the same each time the air pump is used. Hence once the correct airflow is determined the valve should not require any further adjustment.

9. Pump air through the apparatus for 15min.
10. Remove the top, pear shaped flask, with bubbler still attached.
11. Add 0.01N NaOH to burette containing a 40mm glass funnel.
12. Record the initial volume (litre) of 0.01N NaOH in the burette.
13. Turn the plastic stopcock in the bottom of the burette to add the 0.01N NaOH drop by drop into the 2 necked flask until the appearance of the green colour originally produced in the flask in step 4.
Record the final volume (titre) of 0.01N NaOH.
14. Calculate the amount of free sulphur dioxide (SO₂) in the sample as follows.

$$\text{Free SO}_2(\text{ppm}) = (\text{Final Volume of 0.01N NaOH} - \text{Initial Volume of 0.01N NaOH}) \times 16$$

(Note: ppm = parts per million = milligrams/litre)

For Example:

$$\text{Initial Volume of 0.01N NaOH} = 12.42\text{ml}$$

$$\text{Final Volume of 0.01N NaOH} = 15.60\text{ml}$$

$$\text{Free SO}_2 = (\text{Final Volume} - \text{Initial Volume}) \times 16$$

$$= (15.60 - 12.42) \times 16$$

$$= 3.18 \times 16$$

$$= 50.88 \text{ ppm}$$

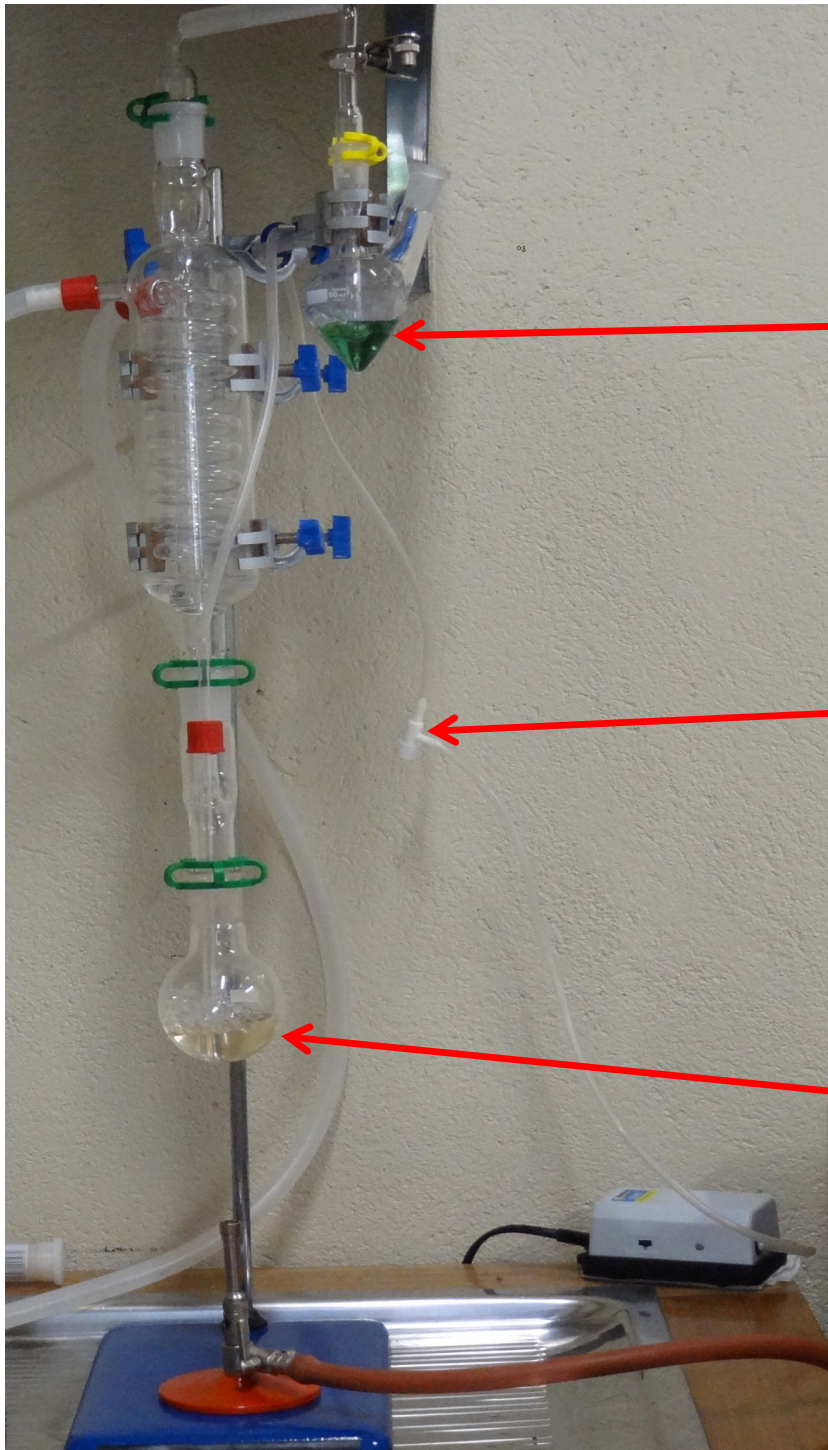
$$\text{Round off} = 51 \text{ ppm}$$

"Bound" Sulphur Dioxide

1. After determining the 'free' sulphur dioxide, replace the pear-shaped flask, including the contents and bubbler, to its position on the apparatus. Pump air through the sample for 15 minutes while applying heat to the bottom flask containing the sample. Heat to a gentle boil and ensure water is flowing in condenser.
2. Remove the top flask, stop the heating and titrate contents with 0.01N sodium hydroxide from burette as before. Record the titre.
3. To calculate the amount of sulphur dioxide in the sample, multiply the titre by 16. Record as "ppm sulphur dioxide - "bound".

NOTE:

To calculate the total sulphur dioxide content of the sample add the "free" and "bound" values.



10 ml 0.3% Hydrogen Peroxide at 20C

3 drops of indicator

Titrate with 0.01 Sodium Hydroxide until olive green (if needed).

Air flow 1 litre per minute

20 ml wine at 20C

10 ml 25% Phosphoric Acid

Making 0.01 Sodium Hydroxide from 0.1N solution: 10 ml 0.1 added to 90 ml distilled water
Making 0.3% Hydrogen Peroxide from 3% solution: 10 ml 3% added to 90ml distilled water

Measuring Titratable Acid, TA

Method for degassing

Degassing can be achieved by placing the sample in a stoppered buchner flask and connecting the side arm to a vacuum system. An electric vacuum pump can be used but if this is not available then a venturi water pump can be used. if used incorrectly. Venturi water type systems are relatively inexpensive but have the added concern of drawing water back into the sample

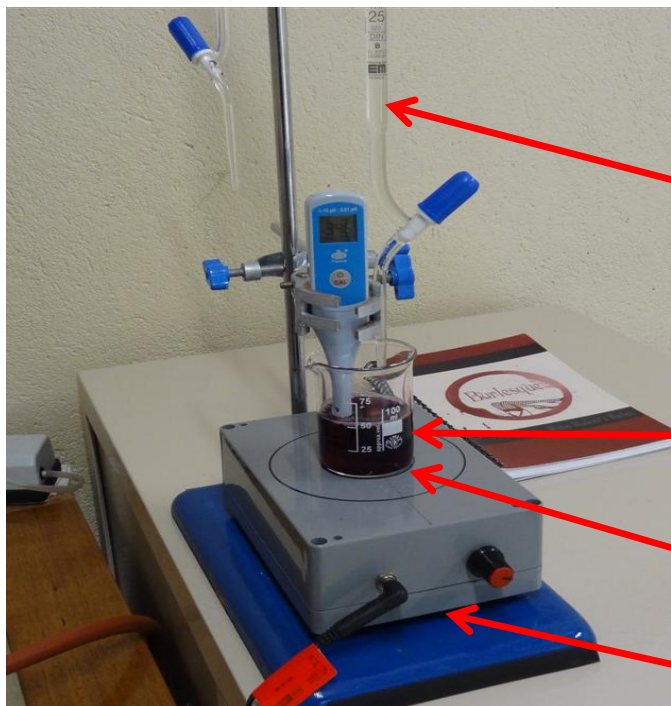
- Pour about 100 mL of wine into a 250 or 500mL buchner flask.
- Fit a rubber stopper securely in the top and connect the side arm to the vacuum system.
- Gently shake the flask for about 2 to 3 minutes under vacuum.
- If a venturi system is used it is important to disconnect the vacuum hose to the flask before turning the water flow off. This will prevent the drawing of water back into the flask.

Alternatively boil wine for 5 seconds in a microwave oven and cool it

Procedure Using a pH Meter

1. Calibrate the pH meter.
2. Fill the burette with 0.1 N NaOH.
3. Add sufficient water to a 250 mL beaker to ensure that the pH electrode will be adequately covered when immersed in the water. Approx. 50-60 even 100 ml will be okay.
4. Rinse the pH electrode with distilled water and gently dry. Discard the rinse waste.
5. Lower the electrode into the beaker of distilled water and position it such that it does not touch the bottom or sides of the beaker.
6. Pipette 10.0 mL of the degassed wine or juice into the beaker
7. Record the initial burette reading.
8. Titrate, with constant and gentle stirring, the solution in the beaker with NaOH until the pH of the solution reaches pH 8.2.
9. Record the final burette reading and calculate the difference between the initial and final readings.
10. Multiply the value by 0.75 This is called the titre value.

eg. $23\text{ml} - 15\text{ml} = 8 \cdot 0.75 = \text{TA of } 6 \text{ g/l}$



0.1 NaOH, Sodium Hydroxide solution

pH meter electrode submerged

10 ml degassed wine or juice

Distilled water to cover pH meter electrode

Magnetic stirrer