

TEST FOR "FREE" AND "BOUND" SULPHUR DIOXIDE

- RANKINE ASPIRATION METHOD

METHOD 1: "FREE SULPHUR DIOXIDE"

1. Remove 0.3% Hydrogen Peroxide from the refrigerator.
2. Use a 10ml volumetric pipette to transfer 10ml of 0.3% H₂O₂ to the 2 - necked, pear shaped flask.
3. Add 3 drops of mixed indicator. The solution may then change to a purple colour. Therefore add one or two drops of 0.01N NaOH in order to adjust colour to an olive green. Alternatively, the solution may naturally change to a green colour. In this case no addition of 0.01 N 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.
4. Adjust temperature of wine sample to 20°C. Use a 20ml volumetric pipette to transfer a sample of wine to the 50 ml Round bottom flask.
5. Now add 10ml of 25% phosphoric acid using a 10 ml volumetric pipette also to the 50ml Round bottom flask.

Note : The 10ml of 25% Orthophosphoric acid may be added directly to the 50ml Round bottom flask by resting the tip of the 10ml vol. pipette on the top of the Air bleed tube and allowing a small space in the top of the tube between the tip of the pipette and the inner wall of the air bleed tube for the air to escape, as the acid is entering the tube. The nylon nut of the swan neck adapter must not be tightened at this stage since the acid may not flow into the 50ml flask. Once 10ml of acid has been transferred to the 50ml flask, immediately finger tighten the nylon locknut (the lock nut must be tightened to be finger tight and not tightened further or the glass thread will be broken) Now check that all parts of the Rankine Apparatus are firmly connected and sealed.

The advantage of this procedure is that as soon as the acid is added to the sample of wine, etc SO₂ is liberated or discharged. The alternative procedure is to add the acid to the 50ml flask and then connect the swan neck adapter then connect to the condenser. During the time taken for this to occur, SO₂ may be lost to the atmosphere, thereby affecting the result.

6. Connect tubing from air pump assembly to the air bleed tube. 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 airflow can be measured or more simply the valve on the air pump assembly adjusted so that the air flow into the air bleed tube is reasonably vigorous. In order to check that the airflow is satisfactory, a Standard SO₂ Solution of 60 ± 2ppm is used in place of the wine sample to obtain 3 similar free sulphur results The flow rate can therefore be verified without the extra expense of the additional equipment. 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. Conversely the water vacuum pump will draw varying amounts of air through the apparatus under vacuum due to the variations in water pressure each time the tap is opened.

7. Pump or draw air by vacuum through the apparatus for 15 min

8. Remove the top, pear shaped flask, with bubbler still attached.
9. Add 0.01N NaOH to burette containing a 50 mm glass funnel.
10. Record the Initial Volume (ml) of 0.01N NaOH in the burette.
11. Turn the plastic stopcock in the bottom of the burette to add the 0.01 N 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 (litre) of 0.01N NaOH.
12. Calculate the amount of Free Sulphur Dioxide (SO₂) in the sample as follows.
13. Free SO₂(ppm) = (Final Volume of 0.01 N NaOH - Initial Volume of 0.01N NaOH) x 16
(Note: ppm = parts per million = milligrams/litre)

