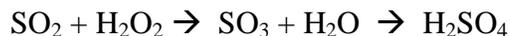


Free SO₂ Determination via Aspiration/Titration Procedure

Free sulfur dioxide (SO₂) from wine or must can be aspirated from an acidified sample by using air as a “purging” gas. The SO₂ driven out of the wine sample by the addition of phosphoric acid is carried by the air aspiration process into a hydrogen peroxide (H₂O₂) trap. When the SO₂ in the air stream bubbles through the hydrogen peroxide it immediately reacts and forms sulfuric acid:



The volume of 0.01 N NaOH (sodium hydroxide) needed to titrate the sulfuric acid product back to a neutral end point is measured and used to estimate free SO₂ levels in the original volumetric sample of wine. A high rate of air aspiration (1.0 L/min) is required for complete recovery of the free SO₂ during the specified aspiration interval (15 min). Typical apparatus used to perform the procedure is depicted in Figure S-1.

Aspiration Procedure:

- a. Make 0.3% H₂O₂: Add 10 ml 3% H₂O₂ to 100 mL flask; top with distilled water.
- b. Add approximately 10 ml of 0.3% hydrogen peroxide and 4 drops of SO₂ indicator solution to the impinge flask (see Figure S-1).
- c. Add 1-2 drops of 0.01 N NaOH to the hydrogen peroxide solution until color is indicative of a neutralized condition (i.e., changes from purple to green).
- d. Secure tubing connections between the wine sample flask and the H₂O₂ trap.
- e. Add precisely 20 ml of wine to the sample flask using a volumetric pipette.
- f. Add 10 ml of 25% phosphoric acid to the sample flask.
- g. Immediately, insert the air bubbler into the sample flask and turn-on the air pump.
- h. At this point, one should observe bubbling in both the sample flask and the H₂O₂ trap (i.e., impinger flask). Contents of the H₂O₂ trap will quickly change color from green to purple as sulfuric acid byproducts begin to accumulate.
- i. Record the start time and allow aspiration to proceed for 15 minutes. After 15 minutes, turn-off air pump.
- j. Use wash bottle to rinse H₂O₂ bubbler residue into the impinger flask.
- k. Remove the impinger flask and titrate from purple back to green indicator color.
- l. Calculate free SO₂ (PPM) as follows: $\text{SO}_2 \text{ (mg/L)} = (\text{ml NaOH}) * 16$
i.e., $(\text{ml NaOH})(\text{N NaOH eq./L})(64 \text{ g/mol})(0.5 \text{ mol/eq.})(1000 \text{ mg/g}) / (\text{ml wine})$

Reagents:

0.01 N NaOH (sodium hydroxide base) titration solution
SO₂ indicator solution (ValleyVintner.com) (purple=acidic vs. green=neutralized)
25% phosphoric acid solution
0.3% hydrogen peroxide solution (1:10 dilution of generic “drug store” 3% H₂O₂)

Equipment:

10 ml graduated cylinder (phosphoric acid); 20 ml volumetric pipette (wine sample)
100 ml volumetric flask; 100 ml volumetric flask
Apparatus depicted in Figure S-1.

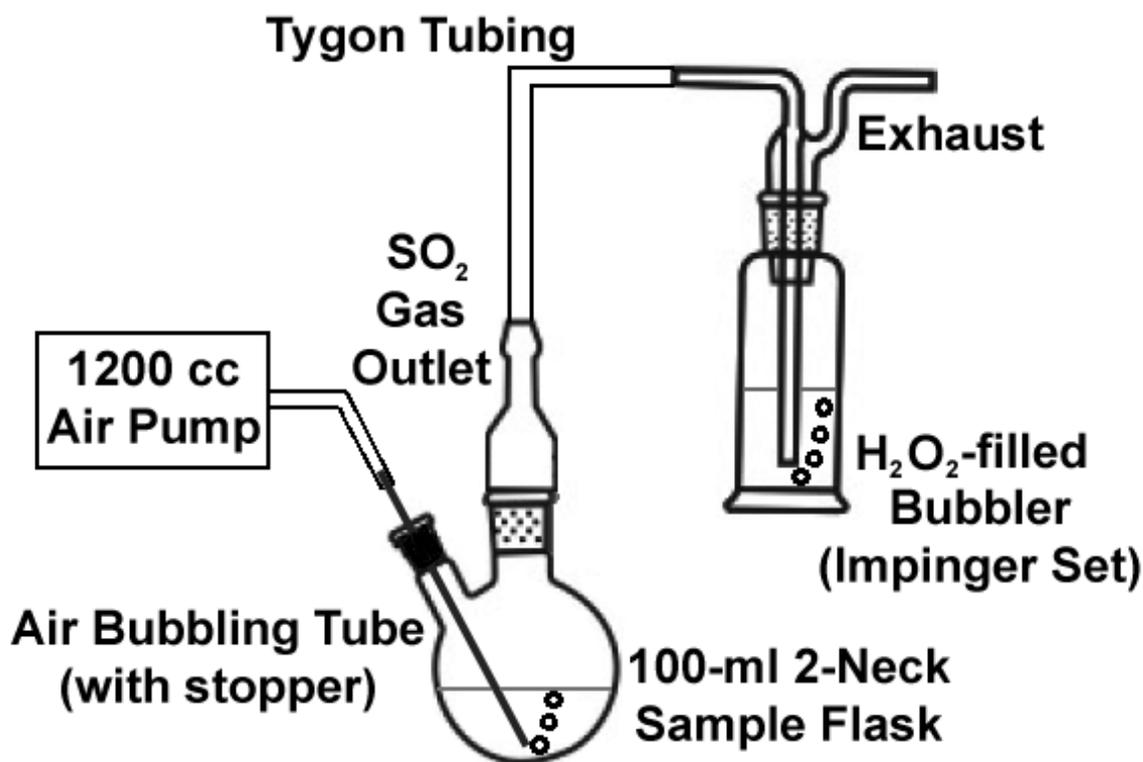


Figure S-1.

Pump forces air through acidified wine via the air bubbling tube in the sample flask. Free SO₂ volatilized by the addition of 25% phosphoric acid is driven via the forced air stream into the hydrogen peroxide solution in the impinger set. SO₂ in air stream mixes with hydrogen peroxide molecules to form titratable sulfuric acid. The amount of sulfuric acid, thus formed, is indicative of the amount of free SO₂ in the wine sample.