TOTAL SO₂ BY AERATION-OXIDATION

Equipment
- Aeration-Oxidation apparatus, including
- Round-bottom side arm flask
- Impinger assembly
- Graham condenser
- Heating mantle
- Vacuum aspirator or other vacuum source
- Ice bath heat exchanger to chill condenser water
- 20mL Volumetric Pipet, Class A
- 10mL Serological Pipet
- 10mL Buret Assembly
- Safety Pipet Bulb

Reagents
- 3% Hydrogen Peroxide, pH adjusted to 5.7
- SO₂ Indicator (Methyl Red + Methylene Blue in 50% Ethanol)
- 0.01N Sodium Hydroxide
- 25% Phosphoric Acid CAUTION: CORROSIVE

Procedure
- Assemble the apparatus, routing the cooling water for the condenser through the ice-bath heat exchanger, and connecting the round-bottom flask to the bottom of the condenser and the impinger tubing to the top of the condenser.
- Fill an impinger bottom to the 10mL mark with 3% Hydrogen peroxide. Add 3 drops of the indicator. (Solution should be grey to grey-green in color. If not, adjust with dilute NaOH or HCl. If too purple - use NaOH; too green - use dilute HCl.)
- Place the impinger bottom on the impinger top.
- Pipet exactly 20mL of sample into 100mL round bottom flask through the side arm. Pipet ca. 10mL 25% phosphoric acid into flask with sample. Add a few boiling chips to the flask to prevent bumping. Place a bubbler/stopper into flask.
Turn on water to aspirator or turn on vacuum pump, aspirate for 10 minutes at a flow of 1000 mL/min.
While aspirating sample, prepare a second impinger bottom as above.
Turn off vacuum. Remove impinger bottom, blowing out any drops left into impinger top. Set this impinger bottom aside.
Place the second impinger bottom on the impinger top. Apply power to the heating mantle. When the solution begins to boil, start aspiration as before. Aspirate for 15 minutes.
While the second aspiration is proceeding, titrate the hydrogen peroxide solution in the first impinger bottom with 0.01N NaOH to a grey to grey-green color. Record the value.
When the second aspiration is finished, remove the impinger bottom as before and titrate as before. Record the value.

Calculations
- Free SO$_2$ (ppm) = N NaOH x mL NaOH x 1600 (for the first titration)
- Bound SO$_2$ (ppm) = N NaOH x mL NaOH x 1600 (for the second titration)
- Total SO$_2$ (ppm) = Free SO$_2$ + Bound SO$_2$

If N of NaOH is 0.01, then: SO$_2$ (ppm) = mLs NaOH x 16

Notes
- If only total SO$_2$ is required, the first aspiration can be omitted. After putting the sample and H$_3$PO$_4$ in the side-arm flask and installing a bubbler/stopper, apply heat and begin aspirating when sample begins to boil. Aspirate for 15 minutes. Titrate as above.
- Total SO$_2$ (ppm) = N NaOH x mL NaOH x 1600
- **Standardize NaOH frequently. Store Hydrogen Peroxide in refrigerator. Make sure all tubing connections are tight.

Disposal
- Hydrogen peroxide-dispose with water in sink.
- Wine with Phosphoric acid-add 5 mL of Kolor-Safe Neutralizer and dispose with water in sink.