



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
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 H₂SO₄.)

Place the impinger bottom on the impinger top.

Pipet exactly 20mls of sample into 100ml round bottom flask through the side arm. Pipet ca. 10mls 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, aspirate for 10 minutes at a flow of ca. 1000ml/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 SO₂ (ppm) = $\frac{N}{1000}$ NaOH x mls NaOH x 1600
(for the first titration)

Bound SO₂ (ppm) = $\frac{N}{1000}$ NaOH x mls NaOH x 1600
(for the second titration)

Total SO₂ (ppm) = Free SO₂ + Bound SO₂

If $\frac{N}{1000}$ of NaOH is 0.01, then: SO₂ (ppm) = mls NaOH x 16

NOTES

If only total SO₂ is required, the first aspiration can be omitted. After putting the sample and H₃PO₄ 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 SO₂ (ppm) = $\frac{N}{1000}$ NaOH x mls 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 mls of Kolor-Safe
Neutralizer and dispose with water in sink.