DETERMINATION of HYDROGEN PEROXIDE CONCENTRATION (20% to 70%)

SCOPE

This method is designed for the determination of hydrogen peroxide in aqueous solutions containing 20% to 70% hydrogen peroxide.

PRINCIPLE

Hydrogen peroxide in a diluted portion of the sample is quantitatively oxidized by titration with a potassium permanganate solution of known strength. Compounds that are oxidized by potassium permanganate under acidic conditions interfere. (Ref: Solvay & Cie., Method FN 1167/01).

REAGENTS

All reagents should be analytical reagent grade, and only deionized water should be used.

A. Potassium Permanganate (KMnO₄): 0.1N-Potassium permanganate is a strong oxidizer; wear gloves and safety glasses. Weigh 3.2 g of KMnO₄ into a 1-liter beaker. Add 500 mL of water and stir until all the KMnO₄ is in solution. Boil for one hour, cool, and filter through a fritted glass crucible into a 1-liter volumetric flask. Dilute to volume and mix well. Store in a dark-colored bottle. Standardize using the method given in Procedure, Item A. below.

B. Sodium Oxalate (Na₂C₂O₄): Sodium oxalate is toxic; wear gloves and avoid breathing dust.

C. Sulfuric Acid (1:3): Wearing gloves and safety goggles, slowly add 50 mL of sulfuric acid (Analytical Reagent Grade 96%) to 150 mL of water in a 250-mL beaker while constantly stirring. Allow the solution to cool to room temperature before using.

PROCEDURE

A. Standardization of Potassium Permanganate (0.1N)

1. Weigh (to the nearest 0.1 mg) about 0.3 g of dry sodium oxalate into a 500-mL Erlenmeyer flask.

2. Add 200 mL of water, 50 mL of H₂SO₄ (1:3), and a few glass beads.

3. Heat the solution to boiling on a hot plate.

4. Remove the flask from heat and add the potassium permanganate solution from a 50-mL Class-A burette until the first appearance of a faint pink color that persists for 30 seconds. Do not let the temperature of the solution in the flask fall below 70°C before the endpoint is reached.

Normality of KMnO₄ = \( \frac{\text{Weight Na}_2\text{C}_2\text{O}_4(2)(1000)}{\text{(mL KMnO}_4\text{(134))}} \)

B. Determination of Hydrogen Peroxide

1. Weigh a 50-mL beaker to the nearest 0.1 mg.

2. Using a Mohr pipette, add to the 50-mL beaker:
   - ± 5 g of sample for 20% to 35% H₂O₂
   - ± 3.5 g of sample for 50% H₂O₂
   - ± 2.5 g of sample for 70% H₂O₂
   - a proportionally larger sample for residual H₂O₂ (e.g., about 20 g for 1% H₂O₂).
Caution: gloves and safety goggles must be worn when handling concentrated peroxide.

3. Immediately reweigh the beaker to the nearest 0.1 mg. Record the gain in weight as W.

4. Transfer the sample to a 500-mL volumetric flask containing about 250 mL of water and 2 mL of H$_2$SO$_4$ (1:3). Thoroughly rinse the beaker into the volumetric flask. Dilute to volume with water and mix well.

5. Pipette 20.0 mL of the solution into a 500-mL Erlenmeyer flask containing 15 mL of H$_2$SO$_4$ (1:3) and 60 mL of water.

6. Add the standardized potassium permanganate solution from a 50-mL Class-A burette until the first appearance of a faint pink color that persists for 30 seconds. Record the volume delivered as V.

**CALCULATION**

\[
\text{% H}_2\text{O}_2 \ (w/w) = \frac{(V)(N)(1.701)(25)}{W}
\]

where: 
- V = mL of potassium permanganate used in titration
- N = normality of potassium permanganate
- W = grams of sample weighed into 50-mL beaker
- 1.701 = weight per milliequivalent of H$_2$O$_2$ x 100
- 25 = dilution factor

**NOTE:** If the proper equipment is available, this titration can be done potentiometrically.

**FIRST AID**

In case of product splashing into the eyes and face, treat eyes first.

**Eye contact:** Flush eyes immediately with water for at least 15 minutes. Call a physician.

**Skin contact:** Immediately flush skin with water while removing contaminated clothing and shoes. Call a physician if irritation persists.

**Inhalation:** Remove the victim from the contaminated area to fresh air. Call a physician in case of respiratory symptoms.

**Ingestion:** Consult with a physician immediately in all cases. DO NOT induce vomiting. If victim is conscious, rinse mouth and give fresh water.

**DANGER**

Hydrogen peroxide solutions are strong oxidizers and corrosive to the eyes, mucous membranes and skin. Consult the SDS for the appropriate Personal Protective Equipment to wear when handling hydrogen peroxide. In case of contact with the eyes, skin or clothing, flush with large amounts of water for 15 minutes. In case of ingestion, sit upright, drink large quantities of water to dilute the stomach contents and seek immediate medical attention. Product in contact with combustible materials may cause fires.
Before using, read Safety Data Sheet (SDS) for this chemical.
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24-hour Emergency Phone Number – 800-424-9300 (CHEMTREC®)

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