

Determination of Hydrogen Peroxide Concentration (0.1% to 5%)

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Technical Data Sheet

Scope

This method is designed for the determination of low levels of hydrogen peroxide (0.1% to 5%) in aqueous solutions.

Principle

Hydrogen peroxide in the sample reacts with excess potassium iodide in the presence of an ammonium molybdate catalyst to produce triiodide ions, which are subsequently titrated with a standard thiosulfate solution.

Reagents

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

A. **Potassium Iodide (10%):** Dissolve 100 g potassium iodide (KI) in 1,000 mL of water.

B. **Acid Mixture:** Dissolve 0.18 g of ammonium molybdate $[(\text{NH}_4)_6 \text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in 750 mL of water. While stirring, slowly add 300 mL of H_2SO_4 (conc.). Wear safety goggles and gloves when handling concentrated H_2SO_4 .

C. **Potassium Iodate Solution (0.1N):** Weigh (to the nearest 0.1 mg) about 3.57g of dried primary standard potassium iodate (KIO_3) and transfer to a 1 liter volumetric flask. Add 400 mL of H_2O , 2 g of sodium hydroxide, and 20 g of potassium iodide. Agitate until dissolution is complete. Dilute to volume and mix well.

D. **Starch Solution (10 g/L):** Weigh 1 g of soluble starch into a 150 mL beaker. While stirring, gradually add about 5 mL of water until a paste is formed. Add the paste to 100 mL of boiling water. Cool and add 5 g of potassium iodide. Stir until dissolution is complete and transfer to a plastic bottle.

E. **Sodium Thiosulfate Solution (0.1N):** Weigh 49.6 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) and transfer to a 2 liter volumetric flask. Add 400 mL of H_2O and agitate until dissolution is complete. Dilute to volume and mix well. Standardize as indicated below:



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TDS XX-122 Revised 1-25-04
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Procedure

A. Standardization of Sodium Thiosulfate (0.1N)

1. Pipette 20.0 mL of potassium iodate solution (0.1N) from a 25 mL pipette into a 500 mL Erlenmeyer flask containing 100 mL of H₂O. Add about 20 mL of potassium iodide (10%) and mix well.
2. Add 25 mL of acid mixture, stopper the flask, and wait five minutes. Using a 50 mL burette, titrate with the sodium thiosulfate solution until the brown triiodide color is nearly dispersed to a pale straw color. Add about 1 mL of the starch solution and titrate until the solution changes sharply from blue to colorless.
3. Record the titration volume and calculate the normality of the sodium thiosulfate solution as shown below. Restandardize every few days, or as often as the solution is needed.
4. Repeat procedure two additional times (3 total) and take the average of the three values as the standardized normality.

$$\text{Normality of sodium thiosulfate solution} = \frac{(\text{g KIO}_3)(\text{mL KIO}_3)}{(\text{mL Na}_2\text{S}_2\text{O}_3)(35.67 \text{ g.L / eq})}$$

B. Determination of Hydrogen Peroxide

1. Weigh 3 g of sample into a 500 mL Erlenmeyer flask.
2. Add 200 mL of water, 20 mL of potassium iodide solution, and 25 mL of the acid mixture.
3. Mix well, stopper, and let stand five minutes.
4. Using a 50 mL burette, titrate with sodium thiosulfate solution (0.1N) until the brown triiodide color has been reduced to a light straw color.
5. Add a few drops of the starch solution (10 g/L) and continue titrating until the color of the solution changes sharply from blue to colorless. Record the volume dispensed as "A".
6. Repeat steps B through E (that is, repeat the titration without the addition of a sample) and record the volume dispensed as "B".

Calculation

$$\text{Hydrogen peroxide \% w/w} = \frac{(A-B)(N)(1.7007)}{\text{Sample Weight}}$$

Where: A = titration volume for sample.
B = titration volume for blank.
N = normality of Na₂S₂O₃

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Storage and Handling

- Store hydrogen peroxide in the original vented container, upright, in a cool, ventilated area where it is protected from damage, or in bulk storage tanks made from approved alloys of aluminum or stainless steel.
- Do not store other chemicals, fuels, or combustible materials near hydrogen peroxide.
- Never return unused hydrogen peroxide to the storage container.
- When empty, rinse all peroxide containers thoroughly with clean water before discarding.
- Use only approved material for pumps, piping, and hoses.

Safety

- Persons working with hydrogen peroxide should be familiar with personal protective equipment, first aid measures and the proper safety and handling procedures. Consult the Material Safety Data Sheet (MSDS) for appropriate information.
- Prevent accidental decomposition by keeping the product free of contaminants.
- Prevent fires by avoiding accidental spills. Water is the preferred method for extinguishing fires in which hydrogen peroxide is present.
- Spills and leaks should be contained, diluted with copious amounts of water and disposed of in compliance with local regulations.
- Hydrogen peroxide storage or handling areas should be equipped with a safety shower, an eyewash station, and a water hose.

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 MSDS 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.

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Before using, read Material Safety Data Sheet (MSDS) for this chemical.

Solvay Chemicals, Inc.

24 hour Emergency Phone Number - 1-800-424-9300 (CHEMTREC®)

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