

Determination of Hydrogen Peroxide (H₂O₂) Residual in Fiber Matrices

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Technical Data Sheet

Scope

This method is suitable for determination of residual hydrogen peroxide in fiber matrices. Materials other than H₂O₂ which are capable of oxidizing iodide will interfere.

Principle

Hydrogen peroxide in the sample reacts with excess iodide in the presence of an ammonium molybdate catalyst to stoichiometrically produce triiodide ions. The triiodide ion concentration is then determined titrimetrically with a standard thiosulfate solution.

Reagents

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

1. Potassium Iodide (10%): In a 2-liter beaker, dissolve 100 g of potassium iodide (KI) in 1000mL of water. Store in a dark glass or opaque, capped bottle.
2. Acid Mixture: In a 2-liter beaker, dissolve 0.18 g of ammonium molybdate ((NH₄)₆ Mo₇O₂₄ • 4H₂O) in 750 mL of water. While stirring, slowly add 300 mL of concentrated H₂SO₄. Store in glass . Wear safety goggles and gloves when handling concentrated H₂SO₄.
3. Sodium Thiosulfate Solution (0.100N): Weigh out 49.63 g of sodium thiosulfate (Na₂S₂O₃ • 5H₂O) and transfer to a 2-liter volumetric flask. Add 400 mL of water and agitate until dissolution is complete. Dilute to volume and mix well. Store in amber or opaque, capped bottle.

The normality of this solution should remain between 0.099N and 0.101N for at least one month. Alternately, standard sodium thiosulfate solutions may be purchased from a laboratory supply company.
4. 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 (KI). Stir until dissolution is complete and transfer to a plastic bottle.



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Procedure

1. Weigh 2 g of the processed fiber sample into a 150-mL Erlenmeyer flask. Record the sample weight to the nearest 0.01 g.
2. Add 50 mL of water and a small magnetic stir bar to the flask containing the sample.
3. Agitate for 15 minutes using a magnetic stirring device. Make sure the slurry is agitated sufficiently to stay suspended during this period.
4. Turn off the stirrer.
5. Add 20 mL of the 10% potassium iodide solution to the flask. Swirl to mix.
6. Add 25 mL of the H₂SO₄/ ammonium molybdate solution to the flask. Swirl to mix.
7. Let the slurry stand for 5 minutes. (Formation of a dark brown color at this point is indicative of H₂O₂ content in the sample.)
8. Resume stirring.
9. Using a 50-mL class-A burette, titrate the flask contents with the standard 0.100N sodium thiosulfate solution until the dark brown triiodide color has been reduced to a yellow hue.
10. Add a few drops of the starch indicator solution to the flask.
11. Resume titrating until the blue color of the solution changes to the natural color of the fiber in water for 30 successive seconds.
12. Turn off the stirrer and let the slurry stand for 2.0 hours. Additional leaching of the H₂O₂ out of the fiber into the solution will be indicated by the reformation of the blue color during this period.
13. Resume stirring and titrate as in "11." above.
14. Record the total volume of sodium thiosulfate dispensed as "A".
15. Repeat steps 2 through 14 with pure water and record the volume of sodium thiosulfate dispensed as "B".

16. Calculation

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

Where: A = titration volume for sample (mL).

B = titration volume for blank (mL).

C = fiber sample weight (g).

N = normality of Na₂S₂O₃ solution.

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Appendix A

Reagent and Laboratory Equipment List

1. Potassium iodide (KI)
2. Ammonium molybdate ((NH₄)₆ Mo₇O₂₄ • 4H₂O)
3. Demineralized water
4. Sulfuric acid (H₂SO₄) (concentrated)
5. Sodium thiosulfate (Na₂S₂O₃ • 5H₂O) or sodium thiosulfate solution (0.100N)
6. Soluble starch
7. 2-liter beakers (2)
8. 500-mL graduated cylinder
9. 2-liter volumetric flask
10. Storage bottles
11. Top-loading analytical balance
12. Magnetic stirring apparatus
13. Small magnetic stir bar
14. 150-mL beaker
15. 50-mL graduated cylinder
16. 20-mL pipette
17. 25-mL pipette
18. 50-mL burette
19. Eyedropper
20. Timer
21. Hot plate
22. 150-mL Erlenmeyer flask

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.

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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.

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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