

Determination of Gasometric Stability of Hydrogen Peroxide (H₂O₂) at 100°C

SCOPE

This method is suitable for measuring the gasometric stability of hydrogen peroxide at strengths less than 75% (w/w).

PRINCIPLE

The sample is placed in a conditioned tube in a silicon oil bath at 100°C. The rate of oxygen evolution is measured with a gas burette.

REAGENTS

Phosphoric Acid, 1:1 – wearing gloves and safety goggles, slowly add 100 mL concentrated Analytical Reagent Grade phosphoric acid to 100 mL water with constant stirring.

APPARATUS

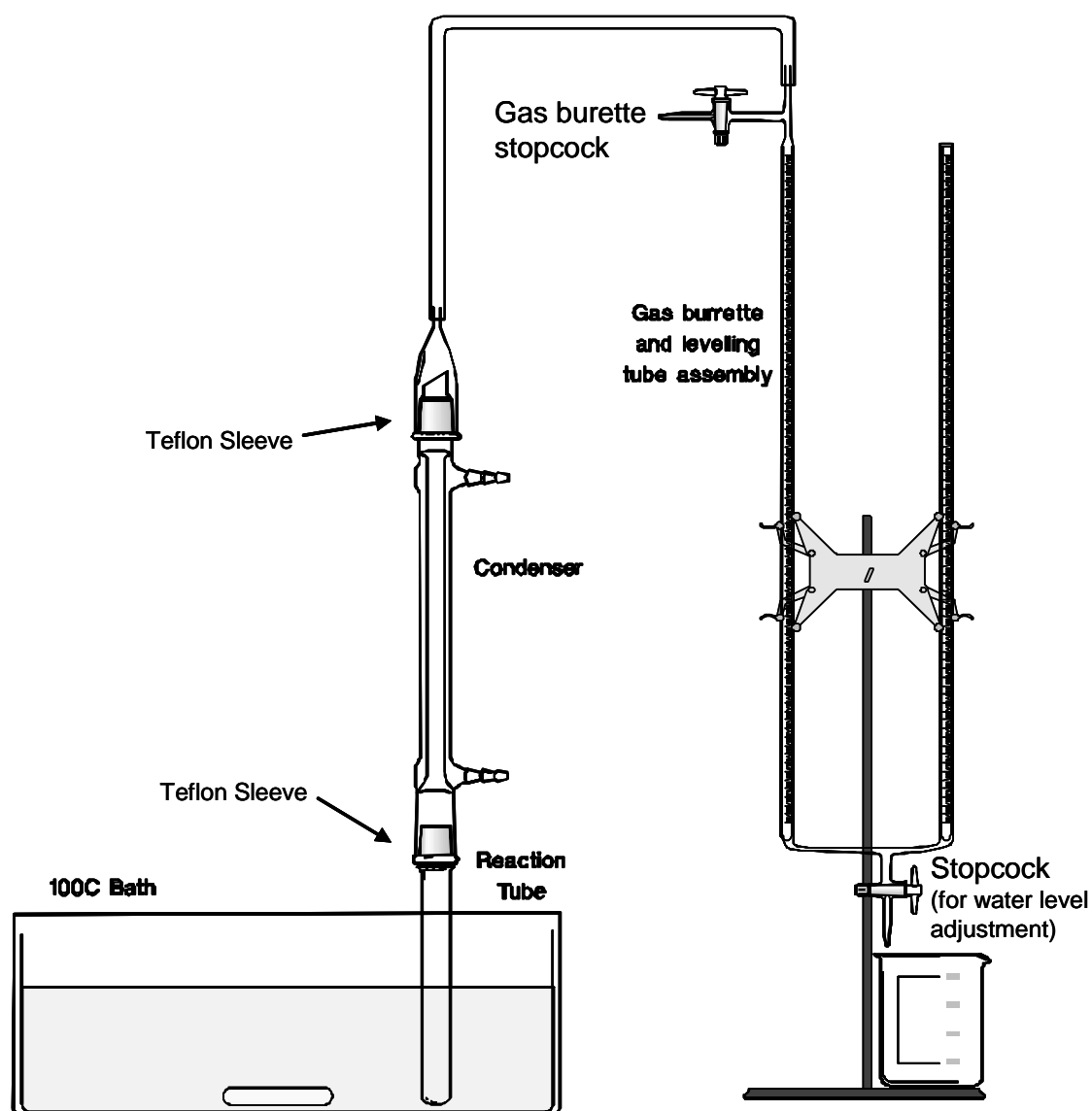
- **Oil Bath** – Capable of maintaining a constant temperature of 100° ± 1°C. The bath should be maintained at an appropriate level with silicone oil. This procedure should be done under a hood in case of an explosion that might occur in the event the H₂O₂ is allowed to evaporate or concentrate.
- **Borosilicate Glass Reaction Tubes** – The tubes have a cone fitting (24/40), an overall length of 6" and external diameter of 1".
- **Borosilicate Glass Condensers** – The condensers are fitted with a socket (24/40) and have an overall length of 18".
- **Gas Burette** – 50-mL capacity marked in 1-mL gradations. A side arm and tap are fitted as shown in Figure 1 (see page 2).
- **Leveling Tubes** – 18" long and 15 mm outside diameter.
- **25-mL pipette** – Fitted with safety bulb.
- **Stopwatch**

Note: Tests must be performed on different grades of hydrogen peroxide using the same reaction tubes. Separate reaction tubes should be kept for each grade of hydrogen peroxide.

All tubes should be kept filled with phosphoric acid solution (1:1). When not in use, store in a dust-free atmosphere.

If the use of an intermediate glass sampling vessel is unavoidable, it should be of borosilicate glass conditioned by treating for 30 minutes with phosphoric acid (1:1), and rinsing well with deionized water before use.

Figure 1
Hydrogen Peroxide
Gasometric Stability Apparatus



To minimize contamination, the pipet for transferring portions to the stability tubes (after conditioning as above) must be kept in a stoppered glass tube when not in use.

Tests must be carried out in duplicate. Reaction tube contents **must not** be allowed to evaporate to dryness.

Do not use this test for H₂O₂ solutions at greater than 75% strength, since an explosion hazard exists.

PROCEDURE

- Turn on the oil bath and adjust to 100° C + 1° C. Also turn on the cooling water to the glass condensers.
- Empty the phosphoric acid (1:1) from the reaction tube. Rinse once with demineralized water and twice with the hydrogen peroxide sample. Caution: gloves and safety glasses must be worn when handling concentrated hydrogen peroxide. Pipet 25.0 mL of sample into the reaction tube and connect the condenser. Lower the apparatus into the preheated oil bath with burette stopcock open. Allow 15 minutes for the conditions to equilibrate.
- Caution: Do not, under any conditions, allow the H₂O₂ sample to evaporate or concentrate. A vapor-phase explosion could occur.
- Adjust the water level in the gas burette to the zero mark by the drop-wise addition of water to the leveling tube. Close burette stopcock and start the stopwatch. Continue the test for 60 minutes. During this time, periodically adjust the water levels in the gas burette and the levelling tube to the same height.
- The water level of the burette and leveling tube should be accurately adjusted and recorded at the end of the test. Let the total volume of oxygen evolved in the gas burette to be "A" mL.

CALCULATION

Calculate the stability of the sample (as mL oxygen liberated per minute from 25.0 mL of sample at 100° C) as follows:

- Gasometric Stability, mL O₂/min/25 mL H₂O₂ = A/60
- Where A = mL oxygen evolved
60 = total minutes of test

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 Safety Data Sheet (SDS) 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 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 fire.

Before using, read Safety Data Sheet (SDS) for this chemical.
Solvay Chemicals, Inc.
24-hour Emergency Phone Number – 1-800-424-9300 (CHEMTREC®)

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