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Determination of Hydrogen Peroxide by Potassium Permanganate Titration

Developed in 1957 by AATCC Committee RA34; reaffirmed 1962, 1968, 1972, 1975, 1979, 2002, 2007, 2012; editorially revised (with title change) 1983, 2010; editorially revised and reaffirmed 1985, 1992; revised 1987 (with title change), 1997.

1. Purpose and Scope

1.1 This test method determines the concentration of hydrogen peroxide (H_2O_2) in aqueous solutions, particularly those used in textile bleaching.

2. Principle

2.1 A specimen is acidified with sulfuric acid and titrated with standardized potassium permanganate solution. The concentration of hydrogen peroxide is calculated using the volume and normality of the permanganate solution used.

3. Terminology

3.1 **bleaching,** n.—elimination of unwanted coloring matter from a textile substrate by oxidative or reductive chemical treatment.

4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The safety precautions are ancillary to the testing procedures and are not intended to be all inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Manufacturers MUST be consulted for specific details such as material safety data sheets and other manufacturer's recommendations. All OSHA standards and rules must also be consulted and followed.

4.1 Good laboratory practices should be followed. Wear safety glasses in all laboratory areas.

4.2 All chemicals should be handled with care. Sodium oxalate, potassium permanganate and sulfuric acid are corrosive. When preparing the dilute solutions from pure or concentrated reagents, use chemical goggles or face shield, impervious gloves, and an impervious apron. CAUTION: Always add acid to water.

4.3 An eyewash/safety shower should be located nearby and a high efficiency particulate respirator with organic vapor cartridge and full facepiece should be readily available for emergency use.

4.4 Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities (e.g., Occupational Safety and Health Administration's [OSHA] permissible exposure limits [PEL] as found in 29 CFR 1910.1000; see web site: www.osha.gov for latest version). In addition, the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) comprised of time weighted averages (TLV-TWA), short term exposure limits (TLV-STEL) and ceiling limits (TLV-C) are recommended as a general guide for air contaminant exposure which should be met (see 11.1).

5. Apparatus

5.1 Filter funnel, fritted glass, fine porosity, 250 mL.

5.2 Filtration flask, 2000 mL.

6. Reagents

6.1 Sodium oxalate ($Na_2C_2O_4$), crystals, CP.

6.2 Sulfuric acid (H_2SO_4), 95-98%.

6.3 Potassium permanganate ($KMnO_4$), crystals.

7. Preparation of Reagents

7.1 Standard 0.100*N* sodium oxalate solution.

7.1.1 Dry at least 7 g sodium oxalate in an oven at 105 ± 1°C (221 ± 2°F) for 4 h and cool in a desiccator.

7.1.2 Weigh 6.7000 ± 0.0002 g sodium oxalate (see 7.1.1) and dissolve in 250-300 mL distilled or deionized water at 70 ± 10°C (158 ± 18°F).

7.1.3 Cool and quantitatively transfer to a 1 L volumetric flask and cover. Allow to stand at least 12 h, then dilute to the mark with distilled or deionized water.

7.2 Sulfuric acid, approximately 20% by volume (see Section 4 for safety precautions).

7.2.1 Slowly add 200 mL 95-98% sulfuric acid to 800 mL water with stirring. Cool to 20 ± 2°C (68 ± 4°F).

7.2.2 Add water to 1 L.

7.3 Potassium permanganate solution 0.588*N* (see 11.2).

7.3.1 Weigh 18.6 ± 0.1 g potassium permanganate and add it to 900 mL water.

7.3.2 Boil the solution for 15 min and cool.

7.3.3 Filter the solution through a filter funnel (see 5.1) into a 1 L volumetric flask. Dilute to the mark with water.

7.3.4 Store solutions of potassium permanganate in brown bottles or otherwise protect the solutions from light.

7.4 Standardization of 0.588*N* potassium permanganate solution. Make three determinations as follows.

7.4.1 Pipette 100 mL 0.100*N* sodium oxalate solution into a 250 mL Erlenmeyer flask and add 10 ± 1 mL 20% sulfuric acid.

7.4.2 Heat to boiling. Remove from heat and immediately titrate with the potassium permanganate solution being standardized, adding the titrant drop by drop at the beginning and at the end of the titration. The end-point is a faint but permanent pink color.

7.4.3 Calculate the normality of the potassium permanganate solution to the nearest 0.001 using the following equation:

$$N_k = (V_o)(N_o)/(V_k)$$

where:

V_o = volume of the sodium oxalate solution used, in mL

N_o = normality of the sodium oxalate solution

V_k = volume of the potassium permanganate solution used, in mL

7.4.4 Average the three normality values and use that average for all calculations.

8. Procedure

8.1 Weigh 10 ± 0.1 g of the aqueous hydrogen peroxide specimen into a 250 mL flask (see 11.3).

8.2 Add 20 ± 1 mL 20% sulfuric acid to the flask (see 7.2). Gently mix by swirling or stirring.

8.3 Titrate the solution with standardized potassium permanganate solution (see 7.3 and 7.4) to a faint pink color which lasts at least 30 s. Record the volume of titrant in milliliters as V_t.

8.3.1 If less than 2 mL of titrant are used, repeat the procedure using either a standardized potassium permanganate solution of known lower normality or a larger specimen.

8.4 Titrate a blank, and record the mL titrant required as V_b.

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