

Test Method for Determination of Hydrogen Peroxide by Potassium Permanganate Titration

Developed in 1957 by AATCC Committee RA34; reaffirmed 1962, 1968, 1972, 1975, 1979, 2002, 2007, 2012, 2016; editorially revised (with title change) 1983, 2010, 2019; 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 0.100N sodium oxalate solution.

7.1.1 Dry at least 7 g sodium oxalate in an oven at $105 \pm 1^\circ C$ ($221 \pm 2^\circ F$) 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 $70 \pm 10^\circ C$ ($158 \pm 18^\circ F$).

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 $20 \pm 2^\circ C$ ($68 \pm 4^\circ F$).

7.2.2 Add water to 1 L.

7.3 Potassium permanganate solution 0.588N (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 0.588N potassium permanganate solution. Make three determinations as follows.

7.4.1 Pipette 100 mL 0.100N 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_r .

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 .

9. Calculations

9.1 Calculate the % H_2O_2 to the nearest 0.01% using the following equation:

% H_2O_2 as 100%

$$= (V_t - V_b)(N_t)(0.017)(100)/(W_s)$$

where:

V_t = volume of titrant in mL

V_b = volume of titrant in mL for blank sample

N_t = normality of the titrant solution

W_s = specimen mass, in g

9.2 If 0.588N KMnO_4 solution is used as the titrant and the specimen mass is 10 g, the equation reduces to:

$$\% \text{H}_2\text{O}_2 \text{ as } 100\% = (0.1)(V_t - V_b)$$

9.3 Using the conditions of 9.2, for reporting % H_2O_2 on other bases, such as 35% or 50%, V_t may be multiplied by the factor $10/B$, where B is the basis expressed as % H_2O_2 . For example, if the basis is 35%,

% H_2O_2 as 35%

$$= (V_t - V_b)(10)/(35) = (V_t - V_b)(0.286)$$

10. Precision and Bias

10.1 Precision.

10.1.1 In 1993 an interlaboratory study included five laboratories, with two oper-

ators each, evaluating two concentrations of hydrogen peroxide in aqueous solution. Two batches of each concentration were prepared. Each operator made duplicate determinations on four separate occasions for each of the two batches, a total of 16 determinations per operator per concentration ($2 \times 4 \times 2$). The data set was analyzed using the ASTM Tex-Pac program (see 11.4).

10.1.2 Analysis showed the residual variances of the two concentrations were different. Accordingly, critical difference tables, with the number of determinations per average shown as n , were prepared for each concentration

10.2 Bias.

10.2.1 Concentration of hydrogen peroxide in aqueous solution can be defined only in terms of a test method. Within this limitation, this method has no known bias in the determination of percent hydrogen peroxide.

Table I—Critical Differences for Two Averages, 95% Probability Level

Hydrogen Peroxide (Grand Average = 0.65%)

n	Single Operator	Within-Laboratory	Between-Laboratories
1	0.03	0.04	0.05
2	0.02	0.03	0.05
4	0.01	0.03	0.04
8	0.01	0.02	0.04

Table II—Critical Differences for Two Averages, 95% Probability Level

Hydrogen Peroxide (Grand Average = 2.88%)

n	Single Operator	Within-Laboratory	Between-Laboratories
1	0.05	0.06	0.11
2	0.04	0.04	0.11
4	0.03	0.04	0.10
8	0.02	0.03	0.10

11. Notes

11.1 Available from Publications Office, ACGIH, Kemper Woods Center, 1330 Kemper Meadow Dr., Cincinnati OH 45240; tel: +1.513.742.2020; web site: www.acgih.org.

11.2 The most widely used standard solution of potassium permanganate is 0.588N. This concentration and 0.1N solution are available from laboratory supply companies.

11.3 If a volumetric aliquot of the aqueous hydrogen peroxide solution is used as the specimen, the specimen mass may be determined from the relationship:

Specimen mass

$$= \text{volume of aliquot (mL)} \times \text{density (g/mL)}$$

The aliquot must be taken at the temperature at which the density is determined. For mill control purposes the density is often disregarded if the bleaching formula remains constant from day to day.

11.4 Available from ASTM International, 100 Barr Harbor Dr., W. Conshohocken PA 19428; tel: +1.610.832.9500; fax: +1.610.832.9555; web site: www.astm.org.