Test Method for Alkali in Bleach Baths Containing Hydrogen Peroxide

Developed in 1960 by AATCC Committee RA34; reaffirmed 1968, 1972, 1975, 1979, 1988, 1989, 2002, 2007, 2012, 2016; revised 1982 (with title change), 1997; editorially revised 1984, 1985, 1987, 2010, 2019.

1. Purpose and Scope

1.1 This test method determines the total alkali content of a bleach bath containing hydrogen peroxide and alkali from any source. The total alkali content is expressed as % sodium hydroxide.

1.2 Alkali in bleach baths may be supplied by sodium hydroxide, sodium silicate, sodium carbonate or by various other alkaline materials, including potassium compounds, ammonia, lime or by peroxygen solids which contain alkaline salts.

1.3 The alkali content of a bleach bath is a critical parameter in determining the rate and extent of bleaching and may also determine the extent to which the textile being bleached is affected by the bleaching process.

1.4 This test method is used for laboratory determinations and for process control.

2. Principle

2.1 A weighed specimen of the bleach bath is titrated with a standardized solution of sulfuric acid to a Phenol Red end-point, or to the pH range 6.8-8.4 on a pH meter. The total alkali, expressed as % NaOH, is calculated based on the weight of the bath.

3. Terminology

3.1 **bleaching,** n.—elimination of unwanted coloring matter from a 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. Sulfuric acid is 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 10.1).

5. Apparatus and Materials

5.1 Potassium acid phthalate ($C_8H_5O_4K$), ACS Reagent.

5.2 Phenol Red (phenol-sulfonphthalein), or 0.06% solution thereof, if a pH meter is not used as an alternative.

5.3 Methanol (CH_3OH) (not needed if 0.06% Phenol Red solution is available).

5.4 Sulfuric acid (0.1*N*) (H_2SO_4), ACS Reagent, 95-98%, or 0.1*N* (see 6.1 and 10.2).

5.5 Sodium hydroxide (NaOH), ACS Reagent.

6. Preparation of Reagents

6.1 Preparation and standardization of 0.1*N* H_2SO_4 (see 10.2).

6.1.1 For each liter of 0.1*N* H_2SO_4 solution to be prepared, weigh 5.5 ± 0.001 g C.P. sulfuric acid and add it to 500 ± 100 mL deionized water with stirring. CAUTION: *Dilution of sulfuric acid is exothermic. Use appropriate safety precautions*. Cover the solution and allow it to cool to 20 ± 1°C (68 ± 2°F). Add the solution to a 1 L volumetric flask, and dilute to mark with deionized water.

6.1.2 Weigh 4.0 ± 0.01 g sodium hydroxide and dissolve it in about 100 mL deionized water in a 250 mL beaker. Transfer the solution to a 1 L volumetric flask, and rinse the beaker 5 times with deionized water, adding the rinses to the volumetric flask. Then dilute to mark with deionized water.

6.1.3 Weigh 20.4080 ± 0.0002 g potassium acid phthalate (National Bureau of Standards sample No. 84) and dissolve it in approximately 100 mL deionized water in a 250 mL beaker. Transfer the solution to a 1 L volumetric flask, and rinse the beaker 5 times with deionized water, adding the rinses to the volumetric flask. Dilute to mark with deionized water.

6.1.4 Using a volumetric pipette, transfer 25 mL of the potassium acid phthalate solution into a 250 mL Erlenmeyer flask. Add 5 drops of 0.06% Phenol Red indicator solution, and titrate with the sodium hydroxide solution to a greenish-yellow end-point, or to pH 7.6 ± 0.8 if using a pH meter to detect the end-point.

6.1.5 Calculate the normality of the sodium hydroxide solution (N_h) to the nearest 0.001 by the following equation:

$$N_h = \frac{(25)(0.1000)}{\text{mL}}$$

where:

mL = the number of mL of sodium hydroxide solution required.

6.1.6 Using a volumetric pipette, transfer 25 mL of the sulfuric acid solution (see 8.1) into a 250 mL Erlenmeyer flask. Add 5 drops of 0.06% Phenol Red indicator solution and titrate with the sodium hydroxide solution to a greenish-yellow end-point, or to pH 7.6 ± 0.8 if using a pH meter to detect the end-point.

6.1.7 Calculate the normality of the sulfuric acid solution to the nearest 0.001 by the following equation:

$$N_s = \frac{(\text{mL})(N_h)}{(25)}$$

where:

N_s = normality of the sulfuric acid solution

mL = number of mL of sodium hydroxide solution

N_h = normality of the sodium hydroxide solution

6.1.8 Adjustment of normality of sulfuric acid solution.

6.1.8.1 If the normality of the sulfuric acid solution is less than 0.0990, discard it and make up a new solution.

6.1.8.2 If the normality of the sulfuric acid solution is greater than 0.1010, adjust it by adding an amount of water cal-

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culated using the following equation:

$$\text{mL } H_2O = \frac{(N)(950)}{0.1000} - 950$$

where:

N = the normality of the sulfuric acid solution

950 = mL sulfuric acid solution after removal of two samples for titration.

6.1.8.3 Re-standardize the adjusted sulfuric acid solution using 6.1.6 and 6.1.7.

6.2 If the indicator solution is prepared in-lab, preparation of 0.06% Phenol Red indicator solution (see 10.2).

6.2.1 Weigh 1.0 ± 0.1 g Phenol Red and dissolve it in 833 mL methanol.

6.2.2 Add 833 mL deionized water.

6.2.3 Cover the solution and stir with a magnetic stirrer until the solution is completely clear.

7. Procedure

7.1 Measure 20 ± 1 mL deionized water into a 250 mL beaker, and add 2-3 drops of Phenol Red indicator solution. If a pH meter is used to detect the end-point, do not add Phenol Red indicator solution.

7.2 Weigh a 10 ± 0.01 g specimen from the bleach bath. Add the specimen to the beaker and mix (see 10.3).

7.3 Titrate the contents of the beaker with 0.1*N* sulfuric acid to a greenish-yellow end-point, or to pH 7.6 ± 0.8 if using a pH meter to detect the end-point.

7.4 Record the number of the 0.1*N* sulfuric acid used to the nearest 0.1 mL.

8. Calculations

8.1 Calculate the total alkali as NaOH to the nearest 0.1% using the following equation:

% Total Alkali, as NaOH

$$= \frac{(\text{mL})(N)(0.040)(100)}{W}$$

where:

mL = the number of mL of the sulfuric acid solution required, as recorded in 6.4.

N = the normality of the sulfuric acid solution, as determined in 8.7.

0.040 = the milliequivalent weight of sodium hydroxide.

W = the mass of the specimen, as determined in 6.2.

8.2 For alternative calculations see 10.4.

9. Precision and Bias

9.1 *Precision*.

9.1.1 In 1995 an interlaboratory study included five laboratories with two operators each, evaluating three concentrations of alkali in aqueous solutions containing 1.15% hydrogen peroxide (H_2O_2). Each operator made three determinations on each concentration. Data were analyzed using the ASTM Tex-pac program (see 10.5).

9.1.2 Analysis showed the residual variances of the three concentrations could be pooled as shown in Table I.

Table I—Critical Differences for Two Averages for the Conditions Noted, 95% Probability Level, % Alkali Concentration

Number of Test Results in Each Average	Single-Material Comparisons		
	Single-Operator Precision	Within-Laboratory Precision	Between-Laboratory Precision
1	0.013	0.027	0.069
2	0.009	0.025	0.069
3	0.008	0.025	0.068
4	0.007	0.025	0.068

9.2 *Bias*.

9.2.1 The results obtained in the interlaboratory study indicate 97.6% average recovery as compared with the calculated values of alkali concentration based on chemical additions. However, the concentration of alkali can be defined only in terms of a test method, and within this limitation this method has no known bias in the determination of alkali concentration.

10. Notes

10.1 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.

10.2 Standardized solutions of sulfuric acid, sodium hydroxide, and Phenol Red indicator are available from various laboratory supply companies in either prepared solution or as concentrates. Standardization of purchased reagent solutions should be verified by the procedures in 6.1.4-6.1.7.

10.3 For process control purposes, specimen volume is often substituted for weight. In this case the % alkali may be in error to the extent of the bleach bath density. If results are to be communicated between installations, specimen mass should be used.

10.4 Simplifications and conversions.

10.4.1 If 0.1*N* H_2SO_4 solution is used, and the specimen mass is 10.0 g, the equation in 8.1 is simplified to:

% alkali as NaOH = (0.04) (mL)

10.4.2 Alkalinity may be defined in terms other than "% as NaOH." Some convenient alternatives, using 0.1*N* sulfuric acid solution, a specimen mass of 10.0 g, and the term "mL" as defined in 8.1, are:

(mL) (2.5) = lb sodium silicate per 100 gal, if the sodium silicate is 10.5% Na_2O

(mL) (2.9) = lb sodium silicate per 100 gal, if the sodium silicate is 8.9% Na_2O

(mL) (0.332) = lb NaOH per 100 gal

10.5 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.

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