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Alkali in Bleach Baths Containing Hydrogen Peroxide

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

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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