Test Method for Alkali in Wet Processed Textiles: Total

Developed in 1975 by AATCC Committee RA34; reaffirmed 1977, 1980, 1986, 1992, 2002, 2007, 2012, 2016; editorially revised 1985, 1990, 2010, 2019; revised (with title change) 1987, 1997.

1. Purpose and Scope

1.1 This test method is used to determine the total alkali content of wet processed textiles. Total alkali can be used to determine washing and/or neutralizing efficiency after certain wet processing steps, particularly bleaching, and can be used as a measure of the suitability of prepared fabric for subsequent dyeing and finishing operations.

2. Principle

2.1 Each specimen is immersed in distilled or deionized water, and the solution is then titrated with a standard acid to a predetermined end point. The percentage of alkali can then be calculated from the amount of acid used and the weight of the specimen.

3. Terminology

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

3.2 **pH,** n.—the negative logarithm of the hydrogen ion activity expressed in gram equivalents per liter used in expressing both acidity and alkalinity on a scale whose values run from 0 to 14 with 7 representing neutrality, numbers less than 7 representing increasing acidity, and numbers greater than 7 representing increasing alkalinity.

3.3 **total alkali,** n.—*in textile wet processing*, residual alkaline materials in a wet processed textile expressed as percent sodium hydroxide on dry weight of the textile.

3.4 **wet processing,** n.—*in textile manufacturing*, a collective term for processes included in preparation, dyeing, printing, and finishing in which the textile material is treated with a liquid, normally water, or with chemicals in solution or dispersion in a liquid.

4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The 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.

4.3 If concentrated sulfuric acid is diluted to prepare the 0.10*N* sulfuric acid (see 6.5), use chemical goggles or face shield, impervious gloves and an impervious apron. Concentrated acids should be handled only in an adequately ventilated laboratory hood. CAUTION: Always add acid to water.

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

4.5 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 13.1).

5. Uses and Limitations

5.1 In this test method all alkalies detected, such as sodium hydroxide, sodium carbonate, sodium bicarbonate, and other alkaline salts, are calculated as sodium hydroxide. The results are reported as total alkali, expressed as NaOH.

5.2 Since distilled or deionized water can contain dissolved carbon dioxide, care should be taken to remove it by boiling water before use.

6. Apparatus and Reagents

6.1 pH meter with 0.1 unit graduations.

6.2 Beakers, glass, 600 mL.

6.3 Burette, glass, 10 mL with 0.10 mL graduations.

6.4 Buffer solution, pH 4.0 and 7.0.

6.5 Sulfuric acid (H_2SO_4), 0.10*N* (see 13.2).

7. Calibration

7.1 Calibrate the pH meter with pH 4.0 buffer solution in accordance with the manufacturer's instructions. Dual calibration instruments should be calibrated to pH 4.0 and pH 7.0.

8. Test Specimens

8.1 Take samples in the dry or wet state.

8.2 Dry cloth. Select two or more representative dried specimens. Place in oven at 100°C (212°F) for 1 h. Cool in a desiccator and weigh to the nearest 0.1 g. Specimens should weigh 5-10 g.

8.3 Wet cloth. Select two or more representative specimens weighing approximately 10-20 g in the wet state.

9. Procedure

9.1 Place each specimen in a 600 mL beaker containing 450-500 mL distilled or deionized water at room temperature and stir vigorously for 1 min. Cover the beaker and keep the specimen immersed for 15 min. Finally, stir the specimen again and insert the pH meter electrode(s), being careful to avoid contact with the specimen.

9.2 Titrate the water and specimen with 0.10*N* H_2SO_4 to a steady (10 s) pH 3.9 endpoint. Add titrant dropwise followed by gentle agitation of the specimen without touching the electrode(s). Read burette to the nearest graduation (see 13.3).

9.3 If the specimen was taken from a wet sample, rinse and dry the titrated specimen to a constant weight (to the nearest 0.1 g) as in 8.2, being careful to collect any large threads that may have unraveled during the titration procedure.

9.4 Titrate a water blank by repeating 9.2 without a specimen present.

10. Calculation

10.1 Compute the percentage total alkali from the titer according to the following formula:

$$X = [(A - B)(0.04)(N)\ 100]/W$$

where:

X = % total alkali as sodium hydroxide (NaOH),
A = volume of acid used for specimen, mL,
B = volume of acid used for blank, mL,
N = normality of sulfuric acid (0.10*N*),
W = weight of specimen.

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10.2 Compute the sample average by using the formula:

$$x = (x_1 + x_2 + \ldots + x_n)/n$$

11. Evaluation

11.1 Total alkalinity is important because of its effect on the subsequent processing operations to which the textile is subjected. Interpretation of test results is usually based on a combination of total alkalinity and pH of the water extracted from the fabric as determined by AATCC TM81, Test Method for pH of the Water Extract from Wet Processed Textiles.

11.2 Total alkalinity quantifies the amount of residual alkali in a specimen, per unit of weight, providing the specimen contains alkali.

12. Precision and Bias

12.1 *Precision*.

12.1.1 In 1993, an interlaboratory study was completed, which included five laboratories, two operators in each, running three determinations per fabric, on four fabrics. No prior assessment was made of the relative level of the participating laboratories in performance of the test method.

12.1.2 Analysis of the data set (5×2×3×4=120 values) yielded components of variance as follows.

Components of Variance	Variance
Laboratories	0.000245
Operators within laboratories	0.000061
Specimens within materials, laboratories, and operators	0.000047

12.1.3 Table I (critical differences) was calculated using the values in 12.1.2.

12.1.4 Differences between two averages of *N* determinations, for the appropriate precision parameter, should reach or exceed the table value to be statistically significant at the 95% probability level.

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

Alkali in Wet Processed Textiles: Total (% weight total alkali as sodium hydroxide)

N	Single Operator	Within Laboratory	Between Laboratory
1	0.0190	0.0288	0.0509
2	0.0134	0.0230	0.0491
4	0.0095	0.0209	0.0481
8	0.0067	0.0198	0.0477

12.2 *Bias*.

12.2.1 The values of the total alkali in wet processed textiles can only be defined in terms of a test method. There is no known bias in the determination of this property by this test method. During this study, no determination of the true value was made by an independent, referee analytical method for the purpose of establishing presence or absence of bias.

13. Notes

13.1 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.

13.2 For method of preparation of standard acid refer to W. W. Scott, *Standard Methods of Chemical Analysis*, 6th Ed., Van Nostrand, New York, 1962, p1343.

13.3 If preferred, titration may be made with a suitable colorimetric indicator such as methyl orange (pH 3.1-4.4). The precision may be less with this method than with a good pH meter.

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