

Test Method for pH and Total Alkali in Wet Processed Textiles: Combined

Foreword

This test method was written as an alternative to AATCC TM81 and AATCC TM144 to offer a combined method for performing these tests on a single specimen (see 14.1).

1. Purpose and Scope

1.1 This test method is used to determine the pH and total alkali content of wet processed textiles. pH and 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.

1.2 To make a quantitative determination, the chemicals which influence pH must be removed from the textile specimen, collected as a water extract and then accurately measured by a pH meter.

2. Principle

2.1 Each specimen is immersed in boiling, distilled or deionized water. The specimen is boiled for an additional time, then covered and allowed to cool to room temperature. The pH of the bath is measured, then titrated with an acid to a predetermined endpoint. The percentage of total alkali on the textile can then be calculated from the amount of acid used and the weight of the specimen.

3. Referenced Documents

NOTE: Use current versions of all publications unless otherwise specified.

3.1 AATCC TM81, Test Method for pH of the Water-Extract from Wet Processed Textiles (see 14.2).

3.2 AATCC TM144, Test Method for Alkali in Wet Processed Textiles: Total (see 14.2).

3.3 W. W. Scott, *Standard Methods of Chemical Analysis*, 6th Ed., Van Nostrand, New York, 1962, p1343.

4. Terminology

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

4.2 **pH**, n.—the negative logarithm of the effective hydrogen ion concentration or hydrogen ion activity in gram equivalent

per liter used in expressing both acidity and alkalinity on a scale with values from 0 to 14, with 7 representing neutrality, numbers less than 7 representing increasing acidity and numbers greater than 7 representing increasing alkalinity.

4.3 **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.

4.4 **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.

5. 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. Users MUST consult manufacturers for specific details such as safety data sheets and other recommendations. Consult and follow all applicable OSHA standards and rules.

5.1 Follow good laboratory practices. Wear safety glasses in all laboratory areas.

5.2 Handle all chemicals with care.

5.3 If concentrated sulfuric acid is diluted to prepare the 0.02N sulfuric acid (see 6.5), use chemical goggles or face shield, impervious gloves and an impervious apron. Handle concentrated acids only in an adequately ventilated laboratory hood. CAUTION: Always add acid to water.

5.4 Ensure an eyewash/safety shower is located nearby and a high-efficiency particulate respirator with a full facepiece is readily available for emergency use.

5.5 Control exposure to chemicals used in this procedure 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 910.1000; 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 av-

erages (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 14.3).

6. Uses and Limitations

6.1 pH and total alkali can be used to determine the suitability of wet processed textiles for subsequent dyeing and finishing operations or to evaluate the washing and/or neutralizing efficiency following any wet processing operation.

6.2 In this test method all alkalis 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.

7. Apparatus and Reagents (see 14.4)

7.1 pH meter, 0.1-unit graduations.

7.2 Beakers, glass, 600 mL.

7.3 Burette, glass, 10 mL, 0.10 mL graduations.

7.4 Buffer solutions, pH 4.0, 7.0, 10.0, or others as needed.

7.5 Sulfuric acid, 0.02N H₂SO₄ (see 3.3 for method of preparation of standard acid).

8. Calibration

8.1 Calibrate the pH meter with pH 4.0 buffer solution in accordance with the manufacturer's instructions. For dual calibration instruments, use pH 4.0 buffer and pH 7.0 or pH 10.0 buffer.

9. Specimens

9.1 Specimens may be taken dry or wet.

9.2 Dry cloth. Select two (2) 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.

9.3 Wet cloth. Select two (2) or more representative specimens weighing approximately 10-20 g in the wet state.

9.4 If the mass per unit area of the fabric is excessively low, or difficult to wet, cut the specimen into small pieces to promote better immersion in the distilled water.

10. Procedure

10.1 Since distilled or deionized water can contain dissolved carbon dioxide, take care to remove it by boiling before use. Boil 450-500 mL of distilled water in a 600-mL beaker at a moderate rate for 10 min. Place each specimen in a separate beaker, cover with a watch glass, and continue boiling for an additional 10 min.

10.2 Allow the covered beaker and contents to cool to room temperature. Stir the specimen, then insert the pH meter electrode(s), being careful to avoid contact with the specimen. Determine the pH of the bath using a pH meter operated according to manufacturer's instructions.

10.3 Titrate the water and specimen with 0.02N sulfuric acid (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 14.5 and 14.6).

10.4 If the specimen was taken from a wet sample, rinse and dry the titrated specimen to a constant weight (as in section 9.2) being careful to collect any large threads that may have unraveled during the titration procedure. Weigh to the nearest 0.1 g.

10.5 Titrate a water blank by boiling (10 min.) and cooling 450-500 mL of distilled water, then titrating as in 10.3, without a specimen present.

11. Calculation

11.1 Calculate the percentage of total alkali from the titer using Eq. 1.

$$X = [(A-B)(0.04)(N) 100]/W \quad (\text{Eq. 1})$$

where:

X = % weight total alkali as sodium hydroxide (NaOH);

A = Burette reading of specimen;

B = Burette reading of water blank;

N = Normality of sulfuric acid (0.02N);

W = Weight of specimen.

11.2 Calculate the mean percentage of total alkali for all specimens.

12. Evaluation

12.1 pH and total alkali of wet-processed textiles depend on the chemical treatment previously given the textile. For example, the pH and alkali would normally be higher after caustic boiling than after bleaching.

12.2 pH and total alkali content are important because of their effect on the subsequent processing operations to which

the textile is subjected. Textiles with high pH and/or alkali values may exhibit yellowing tendencies, exhibit shade changes, or alter the exhaustion and fixation of dyestuffs, and could produce a decrease in the cure of resin finishes or exhaustion of softeners.

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

13. Precision and Bias

13.1 *Precision.* Three (3) operators in a single laboratory participated in an intralaboratory study to measure pH and alkalinity using this method. All operators measured 6 different fabrics and repeated each test 2 times, on separate days. Table I gives the within-lab precision values from this study. Table II provides mean and standard deviation values for each fabric, for reference.

13.2 *Bias.* The true values of pH and alkalinity can only be defined in terms of a test method. Within this limitation, this test method has no known bias.

14. Notes

14.1 A single-laboratory study (3 technicians and 6 fabrics) was conducted in 2017. In this study, all three (3) methods were compared. The data from this study indicated that the pH determined by AATCC TM81 was

slightly different from the pH determined by this combined method. Specimen weight and water volumes are different for AATCC TM81 and AATCC TM144. This combined method uses parameters similar to those of AATCC TM144; this may be the source of variations in pH results between AATCC TM81 and this method.

14.2 Available from AATCC, PO Box 12215, Research Triangle Park NC 27709, USA; +1.919.549.8141; ordering@aatcc.org; www.aatcc.org.

14.3 Available from Publications Office, ACGIH, Kemper Woods Center, 1330 Kemper Meadow Dr., Cincinnati OH 45240; +1.513.742.2020; www.acgih.org.

14.4 For potential apparatus, reagents or materials sources, visit the AATCC Buyer's Guide at www.aatcc.org/bg. AATCC provides its Corporate members the option to their items and services. AATCC does not qualify, or in any way approve, endorse or certify that any of the listings meet the specifications in its standards.

14.5 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.

14.6 An auto-titrator may be used if it has been established by the user that its accuracy is equivalent.

15. History

15.1 Developed in 2019 by AATCC Committee RA34.

Table I—Precision Values

n	Operators	pH (Within Lab)	Alkalinity (Within Lab)
1	1	0.31044	0.02478
1	2	0.43905	0.03505
1	3	0.62091	0.04956
2	1	0.25401	0.01752
2	2	0.31111	0.02478
2	3	0.43997	0.03504
3	1	0.12701	0.01011
3	2	0.17958	0.01429
3	3	0.25401	0.02021
4	1	0.11173	0.00876
4	2	0.15800	0.01239
4	3	0.22345	0.01752
5	1	0.09838	0.00970
5	2	0.13913	0.01372
5	3	0.19676	0.01567

Table II—Precision Study Values

Fabric	Mean pH	St Dev	Mean Alk	St Dev
Twill, Khaki	7.1132	0.2175	0.02185	0.00883
Twill, Navy	7.3803	0.1314	0.02596	0.01010
Twill, White	5.9948	0.1728	0.01035	0.00992
Denim, Indigo	10.104	0.0497	0.00512	0.01255
Denim, Black Overdye	9.3405	0.0829	0.1873	0.0294
Knit, White	9.2165	0.1691	0.07803	0.01288