Designation: D2165 – 94 (Reapproved 2012)ε1

Standard Test Method for pH of Aqueous Extracts of Wool and Similar Animal Fibers[1]

This standard is issued under the fixed designation D2165; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

ε[1] NOTE—The terminology section was updated in July 2012.

1. Scope

1.1 This test method covers the determination of the pH of aqueous extracts from wool and similar animal fibers. It is applicable to fibers in any condition—raw wool, scoured wool, sliver, top, yarn, or fabric.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see Section 11.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D123 Terminology Relating to Textiles
D2525 Practice for Sampling Wool for Moisture
D4845 Terminology Relating to Wool
E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

3. Terminology

3.1 For all terminology related to D13.13, refer to Terminology D4845.

3.1.1 The following terms are relevant to this standard: aqueous extract, pH.

3.2 For all other terminology related to textiles, see Terminology D123.

4. Summary of Test Method

4.1 An extract is prepared using distilled water or 0.1 *N* sodium chloride solution at the boil under reflux, or at room temperature with agitation. The pH of the extract is measured electrometrically with a glass electrode.

5. Significance and Use

5.1 The pH values of the extracts give an indication of the acidity or alkalinity of the fiber and its water-soluble impurities. These values are useful in indicating previous processing and in anticipating subsequent performance. For particular purposes, the pH of an extract prepared by one method may be a more informative index than another and as a consequence four optional extraction procedures are included.

5.2 This test method is not recommended for acceptance testing because the between-laboratory precision is relatively poor. In some cases, the purchaser and the seller may have to test a commercial shipment of one or more specific materials by the best available method, even though the method has not been recommended for acceptance testing of commercial shipments. In such a case, if there is disagreement arising from differences in values reported by the purchaser and the seller when using this method for acceptance testing, the statistical bias, if any, between the laboratory of the purchaser and the laboratory of the seller should be determined, with each comparison being based on testing specimens randomly drawn from one sample of material of the type being evaluated.

6. Apparatus and Materials

6.1 All glassware coming in contact with the liquid shall be of a chemical-resistant glass,[3] in which the contacting surfaces have been soaked for two days in 0.1 *N* hydrochloric acid and then rinsed thoroughly with distilled water (see 7.1) until the rinsings have a pH of 6.0 or higher.

NOTE 1—It is desirable but not mandatory that the glassware be reserved for extraction tests only and be filled with distilled water during storage between tests.

6.2 *Apparatus for Extraction at Room Temperature:*

6.2.1 *Erlenmeyer Flasks,* 250-ml, wide-mouth, with ground-glass stoppers.

[1] This test method is under the jurisdiction of ASTM Committee D13 on Textiles and is the direct responsibility of Subcommittee D13.13 on Wool and Felt.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Borosilicate glass has been found satisfactory.

6.2.2 *Laboratory Shaker or Agitator,* with apparatus for attaching the flasks, holding at least three flasks, to provide agitation that will not raise the temperature more than 5.5°C in 2 h.

6.3 *Additional Equipment Needed for Extraction at The Boil:*

6.3.1 *Erlenmeyer Flask,* 500-mL, with ground-glass joint.

6.3.2 *Air Condenser, Glass,* reflux, to fit the flask.

6.3.3 *Tube,* to hold absorbent for acidic and basic gases.

6.3.4 *Glass Stopper,* for flask, equipped with a stopcock and thermometer with a range from 0 to 105°C.

6.4 *pH Meter and Glass Electrode,* conforming to the requirements of Sections 5 and 6 of Test Method E70.

7. Reagents

7.1 *Distilled Water,* having a pH of between 6.2 and 7.0. If not in that range of pH, redistillation is necessary.

7.2 *Sodium Chloride, Standard Solution (0.1 N),* prepared from reagent grade sodium chloride (NaCl) and distilled water having a pH of between 6.2 and 7.0.

7.3 *Anhydrous Calcium Sulfate or Equivalent Absorbent for Acid or Alkaline Gases.*

8. Sampling and Specimen Preparation

8.1 Take a lot sample of raw wool, scoured wool, sliver, top, yarn, or fabrics as specified in the sampling procedure in Practice D2525.

8.2 Select specimens at random from the unconditioned sample, each weighing 10 ± 0.1 g. Cut the fibers of the specimen into lengths of about 5 mm and blend.

9. Number of Specimens

9.1 Take a number of specimens per laboratory sampling unit such that the user can expect at the 95 % probability level that the test result for a laboratory sampling unit will be no more than 0.5 percentage points above or below the true average for the laboratory sampling unit as follows:

9.1.1 *Reliable Estimate of s*—When there is a reliable estimate of s based upon extensive past records in the user's laboratory as directed in the test method, calculate the required number of specimens per laboratory sampling unit using Eq 1:

$$n = (ts/E)^2 \quad (1)$$

where:

n = number of specimens per laboratory sampling unit (rounded upward to a whole number),

s = reliable estimate of the standard deviation of individual observations on similar materials in the user's laboratory under conditions of single operator precision,

t = value of Student's t for two-sided limits, a 95 % probability level, and the degrees of freedom associated with the estimate of v (Table 1), and

E = 0.5 percentage points, the allowable variation.

9.1.2 *No Reliable Estimate of s*—When there is no reliable estimate of s for the user's laboratory, Eq 1 should not be used directly. Instead, specify the fixed numbers of specimens shown in Table 2. These numbers of specimens are calculated using values of s which are listed in Table 2 and which are somewhat larger values of s than are usually found in practice. When a reliable estimate of s for the user's laboratory becomes available, Eq 1 will usually require fewer specimens than are listed in Table 2.

10. Preparation of Extracts

10.1 *Extraction with Boiling Water*— Include an approximately proportionate quantity of any fallout present in each specimen. Transfer each specimen to a separate flask. Cover the fibers with 200 mL of boiling water (see 7.1). Connect the reflux condenser, making certain that anhydrous calcium sulfate absorbent is in the absorption tube. Shake, to complete wetting of the fiber, and heat gently to maintain boiling. Agitate the solution every 10 min by shaking the apparatus. After 30 to 35 min, remove the flask from the heat source, remove the reflux condenser, and stopper the flasks as quickly as possible with a stopper containing a thermometer. Cool the flask and contents in water maintained at 21 ± 2°C, without removing

TABLE 1 Values of Student's *t* for One-Sided and Two-Sided Limits and the 95 % Probability[A]

df	One-Sided	Two-Sided	df	One-Sided	Two-Sided	df	One-Sided	Two-Sided
1	6.314	12.706	11	1.796	2.201	22	1.717	2.074
2	2.920	4.303	12	1.782	2.179	24	1.711	2.064
3	2.353	3.182	13	1.771	2.160	26	1.706	2.056
4	2.132	2.776	14	1.761	2.145	28	1.701	2.048
5	2.015	2.571	15	1.753	2.131	30	1.697	2.042
6	1.943	2.447	16	1.746	2.120	40	1.684	2.021
7	1.895	2.365	17	1.740	2.110	50	1.676	2.009
8	1.860	2.306	18	1.734	2.101	60	1.671	2.000
9	1.833	2.262	19	1.729	2.093	120	1.658	1.980
10	1.812	2.228	20	1.725	2.086		1.645	1.960

[A] Values in this table were calculated using Hewlett Packard HP 67/97 Users' Library Programs 03848D, "One-Sided and Two-Sided Critical Values of Student's *t*" and 00350D, "Improved Normal and Inverse Distribution." For values at other than the 95 % probability level, see published tables of critical values of Student's *t* in any standard statistical text **(1)**, **(2)**, **(3)**, and **(4)**.

TABLE 2 Specimens Required Under Conditions of Unknown Variability in User's Laboratory, pH Units

Names of the Properties	Number of Specimens	Basis[A]
Distilled water at 21°C	5	$s = 0.154$
Distilled water at boil	7	$s = 0.196$
0.1 *N* NaCl solution at 21°C	3	$s = 0.126$
0.1 *N* NaCl solution at boil	3	$s = 0.126$

[A] The values of s in this table are somewhat larger than will usually be found in practice (see 9.1.2).

the stopper. Measure the pH within 10 min after extraction and cooling have been completed, as directed in Section 11.

10.2 *Extraction with Water at Room Temperature*—Take the two specimens, including an approximately proportionate quantity of any fallout present. Transfer each specimen to a separate flask. Cover the fibers with 100 mL of neutral distilled water at 21°C. Then stopper the flask using a glass stopper having a built-in thermometer. Shake vigorously by hand for about 30 s to wet the specimen thoroughly and then agitate mechanically for 2 h at a rate that will not warm the solution above 28°C. Measure the pH as directed in Section 11.

10.3 *Extraction with Boiling 0.1 N NaCl Solution*—Proceed as directed in 10.1 substituting 0.1 *N* NaCl solution for the distilled water. Measure the pH as directed in Section 11.

10.4 *Extraction with 0.1 N NaCl Solution at Room Temperature*—Proceed as directed in 10.2, substituting 0.1 *N* NaCl solution for the distilled water. Measure the pH as directed in Section 11.

11. Procedure

11.1 Immediately before use with the specimen, standardize the pH meter and electrodes as directed in Section 8 of Test Method E70, using standard buffers selected to bracket the expected pH of the extract, at 21°C.

NOTE 2—**Caution:** If calomel electrodes are used in the pH meter, adequate safety precautions need to be taken because calomel (mercurous chloride) is a toxic substance.

11.2 After the meter has been standardized as directed in 11.1, wash the electrodes and sample container repeatedly with distilled water until the indicated pH value no longer changes. This will require at least three changes of water. Remove the drops of liquid hanging from the electrode by touching with absorbent tissue.

11.3 Remove the stopper from the flask for specimen No. 1 and decant enough extract into the sample container to immerse the electrodes 10 mm below the surface of the liquid. Restopper the flask. Agitate the solution with a stirring rod of chemical-resistant glass or by rotating the sample container until the pH reading reaches a steady value. Discard this portion of the extract but do not rinse the electrodes. Disregard the observed pH reading. In the same way, decant and measure the pH value of further portions, without rinsing the electrodes, until two successive portions agree within 0.1 pH unit. Record these values and the average of the two values to the nearest 0.01 unit.

11.4 If the electrodes are mounted in a cell which does not permit agitation, allow the first portion to stand for 3 min and subsequent portions for 1 min before taking a reading and proceed as directed in 11.3.

11.5 Test each of the other specimens as directed in 11.3 above, being certain not to rinse the electrodes between tests.

12. Calculation

12.1 Calculate the average pH of all pairs of the extracts of each specimen as the average of the last two readings and round the average to the nearest 0.1 pH unit.

12.2 Using the results obtained for the first and second specimens, calculate the standard deviation of each specimen calculated to 0.01 pH unit and round to the nearest 0.1 pH unit.

13. Report

13.1 State that the tests were made on specimens prepared as directed in Test Method D2165, and that the pH was measured as directed in Test Method E70. Describe the material or product sampled and the method of sampling used.

13.2 Report the average pH value and standard deviation and state the extraction method. For example:

	Average Value	Standard Deviation
pH at 21°C – distilled water =	— — — —	— — — —
pH at boil – distilled water =	— — — —	— — — —
pH at 21°C – 0.1 *N* NaCl solution =	— — — —	— — — —
pH at boil – 0.1 *N* NaCl solution =	— — — —	— — — —

14. Precision and Bias

14.1 *Summary*—In comparing two averages, the differences should not exceed the following critical differences in 95 cases out of 100 when all of the observations are taken by the same well-trained operator using the same piece of test equipment and specimens randomly drawn from the same sample of material:

Distilled water at 21°C	0.135 pH units for averages of 5
Distilled water at boil	0.145 pH units for averages of 7
0.1 *N* NaCl solution at 21°C	0.143 pH units for averages of 3
0.1 *N* NaCl solution at boil	0.143 pH units for averages of 3

The size of an observed difference is likely to be affected adversely by different circumstances. The true values of the properties tested by Test Method D2165 can be defined only in terms of specific test methods. Within this limitation, the procedures in Test Method D2165 for determining these properties have no known bias. Paragraphs 14.2 and 14.3 explain the basis for this summary and for evaluations made under other conditions.

14.2 *Interlaboratory Test Data*[4]—An interlaboratory test was run in 1967 in which randomly drawn samples of three materials were tested in each of four laboratories. Each laboratory used one operator who tested two specimens of each

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: **D13-1053** .

material. The components of variance expressed as standard deviations were calculated to be the values listed in Table 3.

TABLE 3 Components of Variance as Standard Deviations, pH Units

Names of the Properties	Single-Operator Component	Between-Laboratory Component
Distilled water at 21°C	0.110	0.410
Distilled water at boil	0.140	0.230
0.1 *N* NaCl solution at 21°C	0.090	0.260
0.1 *N* NaCl solution at boil	0.090	0.200

NOTE 3—Since the interlaboratory tests included only four laboratories, between-laboratory precision data should be used with special caution.

NOTE 4—The tabulated values of the critical differences should be considered to be a general statement, particularly with respect to between-laboratory precision. Before a meaningful statement can be made about two specified laboratories, the amount of statistical bias, if any, between them must be established, with each comparison being based on recent data obtained on randomized specimens from one sample of the material to be tested.

14.3 *Bias*—The true values of the properties listed in Table 3 and Table 4 can only be defined in terms of specific test methods. Within this limitation, the procedures in Test Method D2165 for determining those properties have no known bias.

TABLE 4 Critical Differences[A] for the Conditions Noted, pH Units

Names of the Properties	Number of Observations in Each Average	Single-Operator Precision	Between-Laboratory Precision
Distilled water at 21°C	1	0.305	1.18
	2	0.216	1.16
	4	0.152	1.15
	8	0.108	1.14
Distilled water at boil	1	0.388	0.746
	2	0.274	0.694
	4	0.194	0.666
	8	0.137	0.652
0.1 *N* NaCl solution at 21°C	1	0.249	0.763
	2	0.176	0.742
	4	0.125	0.731
	8	0.088	0.726
0.1 *N* NaCl solution at boil	1	0.249	0.608
	2	0.176	0.582
	4	0.125	0.568
	8	0.088	0.561

[A] The critical differences were calculated using $t = 1.960$, which is based on infinite degrees of freedom.

15. Keywords

15.1 animal fibers; pH; and wool

REFERENCES

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