Designation: D 2165 – 94 (Reapproved 2006)

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

This standard is issued under the fixed designation D 2165; 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.

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]

D 123 Terminology Relating to Textiles

D 2525 Practice for Sampling Wool for Moisture

E 70 Test Method for pH of Aqueous Solutions With the Glass Electrode

3. Terminology

3.1 *Definitions:*

3.1.1 *aqueous extract*, *n— in wool testing*, the solution obtained by digesting a material with water or with a sodium chloride solution to dissolve soluble materials.

3.1.2 *pH*, *n—in common usage*, a measure of acidity or alkalinity of a solution, on a logarithmic scale, with neutrality represented by a value of seven, with increasing acidity represented by decreasingly smaller values, and with increasing alkalinity represented by increasingly larger values.

3.1.2.1 *Discussion*—For a technical discussion of pH, including such phenomena as the effect of temperature on pH, see any recognized chemistry text. The pH of textiles is generally determined on aqueous extracts of the textile being tested.

3.1.3 For definitions of other textile terms used in this test method, see Terminology D 123.

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.

[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 *Apparatus for Extraction at Room Temperature*:

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

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 E 70.

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 D 2525.

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.

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)**.

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 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.

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).