



Standard Test Method for Alkali-Solubility of Wools¹

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1. Scope

1.1 This test method covers a chemical procedure for determination of the amount of wool substance soluble in alkali under standard conditions and is applicable to wool in scoured fiber form, or as fiber obtained from yarn or from woven or nonwoven fabric.

1.2 The values stated in inch-pound are to be regarded as standard. The values stated in SI units are provided for information only.

NOTE 1—This test method is applicable to other animal fibers although the level of alkali-solubility may be different from wool. With individual animal fibers, undamaged solubility should be determined before attempting to assess damage on an unknown sample.

1.3 *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.*

2. Referenced Documents

2.1 ASTM Standards:

D 123 Terminology Relating to Textiles²

D 1060 Practice for Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present²

D 1193 Specification for Reagent Water³

E 1 Specification for ASTM Thermometers⁴

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁵

3. Terminology

3.1 Definitions:

3.1.1 *alkali-solubility, n*—in wool, the percent of clean wool that is soluble in a specified alkaline solution under controlled conditions of temperature and time.

3.1.2 *wool, n*—the fibrous covering of the sheep, *Ovis* species.

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² *Annual Book of ASTM Standards*, Vol 07.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.03.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

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

4. Summary of Test Method

4.1 Specimens are maintained at a stipulated constant temperature in a caustic solution for a specified period of time. The percentage of alkali-solubility is calculated from the loss in mass of the specimen.

5. Significance and Use

5.1 Alkali-solubility is an indication of the degree of damage to wool resulting from certain chemical treatments, particularly when test results on the same wool, before such treatment, are available.

5.1.1 Undamaged scoured wool has typical alkali-solubility in the range of 9 to 15 %. Fine, undamaged wool normally will exhibit higher solubility than coarse wool, because of greater surface area per unit mass of fiber.

5.2 This test method is not recommended for use on wool known to have sustained alkali damage.

5.2.1 Alkali-damaged wool has had material solubilized that ordinarily would be included in the alkali-solubility test results.

5.3 Although results in one laboratory cannot usually be verified in another laboratory, this test method is considered satisfactory for acceptance testing because it has been used extensively in the trade for this purpose and because it is the only available method for assessing damage to wool by an alkali solubility procedure. Comparative tests as directed in 5.3.1 are advisable before Test Method D 1283 is used for acceptance testing.

5.3.1 In case of a dispute arising from differences in reported test results when using Test Method D 1283 for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative tests to determine if there is a statistical bias between their laboratories. Competent statistical assistance is recommended for the investigation of bias. As a minimum, the two parties should take a group of test specimens which are as homogeneous as possible and which are from a lot of material of the type in question. The test specimens should then be randomly assigned in equal numbers to each laboratory for testing. The average result from the two laboratories should be compared using Student's t-test for unpaired data and an acceptable probability level chosen by the two parties before testing is begun. If a bias is found, either its cause must be found and corrected or the purchaser and the

supplier must agree to interpret future test results in the light of the known bias.

6. Apparatus

6.1 *Test Tubes*—Glass test tubes 1.5 in. (38 mm) by 7.9 in. (200 mm) with cork stoppers.

6.2 *Constant-Temperature Bath*—A bath equipped to maintain a temperature of $65 \pm 0.5^\circ \text{C}$ throughout.

6.3 *Thermometer*—ASTM Aniline Point Thermometer, having a range from 25 to 105°C and conforming to the requirements for Thermometer 34C as prescribed in Specification E 1.

6.4 *Weighing Bottles*—Glass bottles of approximately 30 to 35-mL capacity, fitted with ground glass covers.

6.5 *Constant-Temperature Drying Oven*, to be maintained at 105 to 110°C , preferably employing a forced draft.

6.6 *Sieves*⁶—No. 100 (150- μm) approximately 1.75 in. (45 mm) in diameter and 1.75 in. (45 mm) high.

6.7 *Hand Cards*.

6.8 *Analytical Balance*—The balance must be capable of weighing to 0.001 g.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.⁷ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

7.3 *1,1,2 Trichloro-1,2,2 Trifluoroethane*⁸

7.4 *Hydrochloric Acid (1 + 120)*—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 120 volumes of water.

7.5 *Sodium Bicarbonate Solution (2.5 g/L)*—Dissolve 2.5 g of sodium bicarbonate (NaHCO_3) in water and dilute to 1 L.

7.6 *Sodium Hydroxide, Standard Solution (0.100 N)*—Prepare and standardize a 0.100 N solution of sodium hydroxide (NaOH) in carbon dioxide-free water, kept free from access by CO_2 from the air, and dilute to 1 L.

7.7 *Working Reference Wool Samples*—Samples of undamaged scoured wool of known alkali-solubility.

8. Sampling

8.1 *Lot Sample*—As a lot sample for acceptance testing, take at random the number of shipping containers directed in an applicable material specification or other agreement between the purchaser and the supplier, such as an agreement to

use Practice D 1060. Consider shipping containers to be the primary sampling units.

NOTE 2—A realistic specification or other agreement between the purchaser and the supplier requires taking into account the variability between shipping containers, between laboratory samples within a shipping container, and between specimens within a laboratory sample so as to provide a sampling plan which at the specified level of the property of interest has a meaningful producer's risk, consumer's risk, acceptable quality level, and limiting quality level.

8.2 *Laboratory Sample*—As a laboratory sample for acceptance testing, take the number of subsamples from each package in the lot sample as directed in an applicable material specification or other agreement between the purchaser and the supplier, such as an agreement to use Practice D 1060 if baled or bagged wool is to be tested.

8.3 *Test Specimens*—As directed in Section 9, determine the number of specimens to be tested from each subsample in the laboratory sample.

9. Number of Specimens per Subsample

9.1 *Control or Non-Critical Testing*—For routine control testing or other non-critical purpose where acceptance testing is not involved, four specimens, randomly chosen from a lot, may be tested as directed in Sections 10 and 11.

9.2 *Acceptance Testing:*

9.2.1 Take a number of specimens per subsample in the laboratory sample, such that the user may expect at the 95 % probability level that the test result for a subsample in the laboratory sample is not more than 2.0 % above or below the true average of the subsample in the laboratory sample (see Table 1). Determine the number of specimens per subsample in the laboratory sample as follows:

9.2.1.1 *Reliable Estimate of v* —When there is a reliable estimate of v based upon extensive past records for similar materials tested in the user's laboratory as directed in the method, calculate the required number of specimens per subsample in the laboratory sample using Eq 1:

$$n = (tv/A)^2 \quad (1)$$

where:

n = number of specimens per subsample in the laboratory sample (rounded upward to a whole number),

v = reliable estimate of the coefficient of variation of individual observations on similar materials in the user's laboratory under conditions of single-operator precision,

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

A = 2.0 % of the average, the value of the allowable variation.

9.2.1.2 *No Reliable Estimate of v* —When there is no reliable estimate of v for the user's laboratory, Eq 1 should not be used directly. Instead, specify the fixed number of 17 specimens per subsample in the laboratory sample. This number of specimens is calculated using $v = 4.2$ % of the average, which is a somewhat larger value of v than is usually found in practice. When a reliable estimate of v for the user's laboratory becomes available, Eq 1 will usually require fewer than 17

⁶ Detailed requirements for these sieves are given in Specification E 11.

⁷ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

⁸ This solvent is available under various trademarks as Refrigerant-113.

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.

specimens per subsample in the laboratory sample.

10. Preparation of Specimens

10.1 Fabric and Yarn:

10.1.1 *Fabric*—Take the required number of 2-g subsamples from different areas of the fabric lot. Cut each subsample into small squares and unravel the fabric to obtain the yarn. Keep the subsamples separate. Reduce the yarn so obtained, or an equal quantity of yarn originally available for test, to fiber form by the use of a set of hand cards. Take care to retain all fibers; treat the fibers as prescribed in 10.3.

10.1.2 *Yarn*—Take the required number of 2-g subsamples from different areas of the yarn lot. Keep the subsamples separate. Reduce the yarn to fiber as instructed in 10.1.1.

10.2 *Fiber*—Loose scoured or processed wool, card sliver or wool top may be used without additional opening; treat the fibers as prescribed in 10.3.

10.3 *Extraction and Carding*—Extract the wool fibers with successive portions of residue-free 1,1,2 trichloro-1,2,2 trifluoroethane at room temperature until evaporation of a 2-mL portion of the extract leaves no visible residue on a watch glass. Air-dry the fiber at room temperature or slightly above, preferably under forced draft. Hand card the wool. Remove gross impurities, such as pieces of skin, burrs, etc., by hand, if present.

10.4 *Lime-Pulled Wool*—Lime-pulled wool requires special treatment. Remove as much lime as possible by physical separation. Treat the wool with HCl (1 + 120) at room temperature for 15 min, using 100 mL of solution per gram of wool. Squeeze, rinse in water, and squeeze again.

10.5 *Buffer Treatment*—Treat the wool to be tested with a buffer solution consisting of NaHCO₃ (2.5 g/L) at room temperature for 15 min, using 100 mL of solution per gram of wool. Squeeze and rinse, with mild agitation, in two successive portions of neutral water. Squeeze the wool again and partially dry at a temperature not exceeding 105° C. Cut the carded wool with scissors into ½ to ¾-in. (10 to 15-mm) lengths, and dry the approximate required mass in the oven at 105 to 110° C. Transfer to a weighing bottle.

11. Procedure

11.1 From each of the subsamples, take one test specimen which, after oven drying, will weigh 1.0 ± 0.1 g. Oven-dry those specimens at 105 to 110°C to constant mass, determined

to the nearest 0.001 g and test them separately. Avoid drying the specimens longer than required to reach constant mass (no further decrease in mass of more than 0.2 % in 15 min of additional drying).

11.2 Use a separate test tube for each specimen to be tested. Into each test tube pour 100 mL of 0.100 *N* NaOH solution and loosely stopper.

11.3 Put the test tubes in the constant temperature water bath and bring to 65°C.

11.4 Immerse each dried and weighed specimen in the 65°C NaOH solution in the test tubes.

11.5 Stir each specimen once as soon as it is placed in the solution, and again 10 min later, to ensure complete wetting-out.

11.6 After 60 ± 0.1 min of immersion of the specimens in the solution, pour the contents of each test tube through separate sieves. Rinse the tubes twice with tap water to remove any adhering particles and to deposit them on the sieves. Rinse the material collected on the sieves for 5 min by means of a stream of ambient tap water running at approximately 3 L/min.

11.7 Place the sieves on blotting paper or toweling to absorb the excess water and partially dry the specimens in the oven. When most of the moisture has been removed, return the specimens to their respective weighing bottles and dry to constant mass.

11.8 Weigh each specimen to the nearest 0.001 g, drying until successive weighings show no further decrease in mass of more than 0.2 % in 15 min of additional drying time.

11.9 Test a reference sample of wool of known alkali-solubility with every twentieth test or every 6 months, whichever comes first, along with the unknown, to verify the operational procedure.

12. Calculation

12.1 Calculate the alkali-solubility, as a percentage of the original mass, according to Eq 2:

$$\text{Alkali-solubility, \%} = (M_1 - M_2)/M_1 \times 100 \quad (2)$$

where:

M_1 = mass of oven-dry specimen before sodium hydroxide treatment, and

M_2 = mass of oven-dry specimen after sodium hydroxide treatment.

12.2 Report results to the nearest 0.1 %.

13. Report

13.1 If all of the individual specimen results fall within $\pm 10\%$ of the average solubility of (1) the control specimens, or (2) the subsample for acceptance testing, accept the test results for the group. If one or more individual specimen results differs by more than $\pm 10\%$ from the group average, test an additional number of specimens tested for the group and report the average of all specimens for that group.

14. Precision and Bias

14.1 *Interlaboratory Test Data*⁹—An interlaboratory test was run about 1963 in which randomly drawn samples of four materials were tested in each of 14 laboratories. Each laboratory used one operator, who tested four specimens of each material. The components of variance for alkali-solubility results expressed as coefficients of variation were calculated to be:

Single-operator component 4.8 % of the average
 Between-laboratory component 12.0 % of the average

⁹The description of the interlaboratory test is based on the information on precision inserted in 1964.

14.2 *Precision*—For the components of variance reported in 14.1, two averages of observed values should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences listed as follows:

Number of Observations in Each Average	Critical Differences, ^{A,B} Percent of the Grand Average for the Conditions Noted	
	Single-Operator Precision	Between-Laboratory Precision
1	13.3	35.8
4	6.9	33.9
8	4.7	33.6
16	3.3	33.4

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

^B To convert the tabulated values of the critical differences to units of measure, multiply the critical differences by the average of the two specific sets of data being compared and then divide by 100.

NOTE 3—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 specific 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 value of alkali-solubility can be defined only in terms of a specific test method. Within this limitation, Test Method D 1283 for measuring alkali-solubility has no known bias.

15. Keywords

15.1 Alkali-solubility and wool

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