Designation: D4963/D4963M – 11

Standard Test Method for Ignition Loss of Glass Fiber Strands and Fabrics[1]

This standard is issued under the fixed designation D4963/D4963M; 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.

1. Scope

1.1 This test method covers primarily the determination of ignition loss of glass fiber textiles. This method applies to glass fiber strands, twisted or untwisted, coated or uncoated; and fabrics, woven, nonwoven, knitted, coated, and uncoated, and chopped strand. This procedure may be applied to other glass textiles where the amount of organic content obtained by ignition loss is required.

NOTE 1—This test method may be used with other glass fiber classifications, such as C or D, but a different ignition temperature and exposure time may be required. In these cases the manufacturer should be consulted for the appropriate ignition conditions.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

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:*[2]

D123 Terminology Relating to Textiles

D578 Specification for Glass Fiber Strands

D7018 Terminology Relating to Glass Fiber and Its Products

3. Terminology

3.1 *Definitions*—For all terminology related to Subcommittee D13.18 on Glass Fibers, see Terminology D7018.

3.2 For definitions of other textile terms used in this test method, refer to Terminology D123. For information on the designation of construction of glass strands, refer to Specification D578.

4. Summary of Test Method

4.1 The organic content on glass fiber textiles is determined by weighing the specimen before and after ignition. The amount of ignition loss on a sample is reported as a percentage of the total mass before ignition.

5. Significance and Use

5.1 This test method is considered satisfactory for acceptance testing of commercial shipments because current estimates of between-laboratory precision are acceptable.

5.1.1 In cases of a dispute arising from differences in reported test results when using this test method 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 results 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 the testing begins. 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 view of the known bias.

5.2 Glass fiber textiles are provided with various sizings or coatings. These provide a protection for the individual fibers, yarns, or fabric that may compose the glass fiber textile as well as compatibility with further finishing requirements. The amount of sizing or coating on glass fiber textiles as determined by this procedure is used for process control.

6. Apparatus and Materials

6.1 *Reel*—A hand- or motor-driven reel having at least a 1-m [1-yd] perimeter. The reel shall be fitted with a traversing mechanism that will avoid bunching the successive wraps, and

[1] This test method is under the jurisdiction of ASTM Committee D13 on Textiles and is the direct responsibility of Subcommittee D13.18 on Glass Fiber and its Products.

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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.

with an indicator of the length wound. A warning bell that will ring at a specified length is recommended. It is advisable that one arm will be collapsible to allow for easy removal of skeins.

6.2 *Muffle Furnace,* capable of maintaining 625 ± 25°C [1157 ± 45°F].

6.3 *Air Circulating Drying Oven,* capable of maintaining a temperature of 105 ± 2°C [220 ± 4°F].

6.4 *Thermometer, or thermocouple,* for muffle furnace capable of registering 625°C with 25°C maximum increments [1157°F with 45°F maximum increments].

6.5 *Analytical Balance,* 200-g capacity, readable to 0.001 g.

6.6 *Weighing Containers.*[3]

6.7 *Hood,* suitable for removing combustion products.

6.8 *Desiccator,* of sufficient size to hold the weighing containers and an efficient desiccant.

6.9 *Tongs,* long handle, heat-resistant.

6.10 *Gloves,* heat-resistant.

7. Safety Hazards

7.1 *Precautions*—Avoid contact with the hot muffle furnace. Use tongs to remove samples. Prescribed safety gloves should be worn when performing high-temperature [over 45°C or 100°F] testing. Place hazard warning safety signs in a conspicuous place. The muffle furnace must be located under a hood suitable for removing combustion products.

8. Sampling and Number of Specimens

8.1 *Lot Size*—A lot is defined as a single shipment of a single type of glass fiber textile. A lot may constitute all or part of a single customer order.

8.2 *Lot Sample*—As a lot sample for acceptance testing, take the number of sampling units of glass fiber textiles directed in an applicable material specification or other agreement between the purchaser and the supplier.

8.3 *Laboratory Sample*—Consider strand packages or fabric rolls or chopped strand quantities of at least 50 g to be a laboratory sample unless otherwise agreed upon between the purchaser and the supplier.

8.4 *Test Specimens*—As test specimens for acceptance testing, proceed as follows:

8.4.1 For strands, take two lengths of strand, each weighing at least 6 g from each package in the laboratory sample.

8.4.2 For fabrics, cut two test specimens at least 103 cm^2 [16 $in.^2$] and weighing at least 6 g from the laboratory sample in such a way that no specimen is closer than one tenth the width of the swatch from the selvage with no two specimens cut perpendicular to the warp containing the same set of warp ends or if cut parallel to the filling, containing the same set of filling picks, and the specimens from different swatches are each taken from a different part of the width of the swatches, with no specimen being taken within 1 m [1 yd] of the very outside of the roll.

8.4.3 For bulk glass textiles, such as chopped strand, take two quantities randomly as test specimens, each weighing at least 6 g from each laboratory sampling unit.

9. Procedure

9.1 Precondition each test specimen by drying for 1 h at 105 ± 2°C [220 ± 4°F], unless otherwise specified. Remove the test specimens from the drying oven and cool in the desiccator for a minimum of 10 min in the standard atmosphere for testing glass textiles.

NOTE 2—Conditioning is often omitted in current lab practices, but must be used to resolve finish level conflicts between purchaser and supplier.

9.2 Precondition the weighing containers by placing the empty containers in the muffle furnace at 625 ± 25°C [1157 ± 45°F]. After 30 min, remove and cool in the standard atmosphere for testing glass textiles for 30 min.

9.3 Weigh the empty container to the nearest 0.001 g. Record this as the tare mass, *T*.

9.4 Identify each container with respect to each test specimen.

NOTE 3—When it is known that no ash residue separates from the specimen during the weighing and igniting process, the specimen is allowed to be weighed separately without the container. When this occurs, *T* equals zero.

9.5 Place the test specimen in the container and weigh to the nearest 0.001 g. Record this as the initial mass, *A*.

9.6 With the test specimen in the container, place in the muffle furnace. Ignite at 625 ± 25°C [1157 ± 45°F] for at least 30 min.

NOTE 4—For fabrics with less than 3 % loss on ignition, some manufacturers have found that ignition time of 10 min is sufficient. In case of dispute, the conditions in 9.6 shall be used.

9.7 Remove the container with specimen residue from the muffle furnace and cool in the desiccator for at least 30 min in the standard atmosphere for testing glass textiles.

9.8 Remove each container and test specimen separately from the desiccator, and immediately weigh to the nearest 0.001 g. Record this as the ignited mass, *B*.

10. Calculation

10.1 Calculate the ignition loss of the glass textile in percent to three significant digits for each specimen using Eq 1:

10.2 Calculate the average ignition loss for each sampling unit and for the lot as directed in an applicable material specification or contract order.

$$\text{Ignition loss, \%} = 100 \times (A - B)/(A - T) \quad (1)$$

where:

A = initial mass of container and specimen before ignition, g,

B = mass of container and glass residue after ignition, g, and

T = mass of container (Note 3).

[3] Porcelain Crucible, Coors No. E-7, or other containers or holders, suitable for exposure to 625 ± 25°C [1157 ± 45°F], have been found satisfactory for this purpose.

11. Report

11.1 Report that the specimens were tested for ignition loss as directed in Test Method D4963. Include the product description.

11.2 Report, for each laboratory sampling unit of glass textile, the average ignition loss.

11.3 Report, for the lot average, the average ignition loss for all laboratory sampling units that were tested.

12. Precision and Bias

12.1 *Summary*—In 95 out of 100 cases when comparing two averages of two determinations each, the differences should not exceed the following amounts when all of the determinations 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 but tested at different times.

Class 1 vinyl-coated glass yarn—0.8 % of the average
Class 2 vinyl-coated glass yarn—1.5 % of the average

Larger differences are likely to occur under all other conditions. This test method has no bias since the true value of ignition loss can only be defined in a specific test method. Paragraphs 12.2-12.4 explain the basis for this summary and for evaluations made under other conditions.

12.1.1 The values in Table 1 are shown in percent of the average. It is expected that the values will also apply to other forms of glass textiles.

12.2 *Interlaboratory Test Data*[4]—An interlaboratory test was run in 1986 in which randomly drawn specimens of a Class 1 vinyl-coated glass yarn and a Class 2 vinyl-coated glass yarn were tested in each of four laboratories. Each laboratory used two operators, each of whom tested two specimens of each material at different times. The components of variance expressed as coefficients of variation are listed in Table 1.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting RR:D13-1078.

TABLE 1 Coefficients of Variation, Vinyl-Coated Glass Yarns, Percent of Average

Material	Single-Operator Component	Within-Laboratory Component	Between-Laboratory Component
Class 1			
Single-Material	0.41	0.91	0.00
Multi-Material	0.41	0.91	0.99
Class 2			
Single-Material	0.77	0.00	0.85
Multi-Material	1.23	0.00	0.85

TABLE 2 Critical Differences for the Conditions Noted, Vinyl-Coated Glass Yarns, 95 % Probability Level, Percent of Average

Number of Observations in Each Average	Single-Operator Precision	Within-Laboratory Precision	Between-Laboratory Precision
Class 1—Single-Material Comparison			
1	1.1	2.8	2.8
2	0.8	2.6	2.6
5	0.5	2.6	2.6
10	0.4	2.5	2.5
Class 1—Multi-Material Comparisons			
1	1.1	2.8	3.9
2	0.8	2.6	3.8
5	0.5	2.6	3.8
10	0.4	2.5	3.7
Class 2—Single-Material Comparisons			
1	2.1	2.1	3.2
2	1.5	1.5	2.8
5	1.0	1.0	2.5
10	0.7	0.7	2.5
Class 2—Multi-Material Comparisons			
1	2.5	2.5	3.4
2	2.0	2.0	3.1
5	1.6	1.6	2.9
10	1.4	1.4	2.8

NOTE 5—The square roots of the components of variance are being reported to express the variability as percent ignition loss rather than as the square of that unit of measure.

12.3 *Precision*—For the components of variance reported in Table 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 in Table 2.

NOTE 6—Since the interlaboratory test included only four laboratories, estimates of between-laboratory precision should be used with special caution.

NOTE 7—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 any two specific laboratories, the amount of statistical bias if any, between them must be established, with each comparison based on recent data obtained on specimens taken from a lot of material of the type being evaluated and nearly homogeneous as possible and then randomly assigned in equal numbers to the two laboratories.

12.4 *Bias*—The procedure in this test method has no bias because the value of ignition loss can be defined only in terms of a test method.

13. Keywords

13.1 glass fabrics; glass textiles; glass yarns; ignition loss