



Designation: D 6652 – 01

Standard Test Method for Determining Fibrous Debris From Nonwoven Fabrics¹

This standard is issued under the fixed designation D 6652; 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 the quantifying of fibrous debris released and generated from fabrics.

1.2 This test method applies to all fabrics used as wiping materials for purposes, such as house-cleaning, cleaning, cleanrooms, spill clean-up or removal, industrial wipes, shop towels, polishing cloths, etc.

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

1.4 *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 6651 Test Method for Determining Rate of Sorption and Sorptive Capacity of Nonwoven Fabrics³

3. Terminology

3.1 *Definitions:*

3.1.1 *extrinsic sorptive capacity, n*—in textile fabrics, the sorptive capacity of a fabric to a specified liquid on a per-unit-area basis under specified conditions.

3.1.1.1 *Discussion*—While extrinsic sorptive capacity is expressed in terms of volume per unit area, intrinsic capacity

has been used to describe capacity in terms of volume per unit mass. By way of example, if a fabric exhibited an intrinsic capacity of 5 mL/g, that mass of fabric would hold 5 mL whether it was part of a 50 g/m² or 200 g/m² fabric. The extrinsic sorptive capacity would, however, be four (4) times higher for the 200 g/m² fabric than for the lighter weight material.

3.1.2 *fibrous debris, n*—in wiping fabrics, fibrous material released from a fabric during actions such as wet cleaning, polishing or wiping processes, under specified conditions.

3.1.3 *sorption, n*—in textile fabrics, a process in which liquid molecules are taken up either by absorption or adsorption, or both.

3.1.4 *sorptive capacity, n*—in textile fabrics, the maximum amount of liquid absorbed and adsorbed under specified conditions.

3.1.5 *wiper, n*—in textile fabrics, fabric swatches used for such actions as housekeeping, cleaning, polishing, spill clean-up or removal.

3.2 For definitions of other terms used in this test method refer to Terminology D 123.

4. Summary of Test Method

4.1 *Releasable Fibrous Debris (F_o)*—A specimen of known dimensions is placed flat in a tray and gently sluiced with water. The resulting suspension is filtered through a membrane filter, and the releasable fibrous debris counted using optical microscopy.

4.2 *Generated Fibrous Debris (F_G)*—After the releasable fibrous debris (F_o) of a specimen has been determined, the same specimen (now devoid of readily releasable fibrous debris) is placed in a jar and shaken for three minutes along with a volume of water equal to twenty times the sorptive capacity of the ply being tested. The resulting suspension is filtered through a membrane filter, and the generated fibrous debris counted using optical microscopy.

¹ This test method is under the jurisdiction of ASTM Committee D13 on Textiles and is the direct responsibility of Subcommittee D13.90 on Executive.

Current edition approved April 10, 2001. Published July 2001.

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

³ *Annual Book of ASTM Standards*, Vol 07.02

4.3 *Simultaneously Determined Releasable Fibrous Debris and Generated Fibrous Debris (F)*—Releasable and generated fibrous debris is determined by either of two procedures: (1) releasable fibrous debris and generated fibrous debris are determined separately and the results added together, or (2) the procedure described for determining generated fibrous debris only is used and the releasable fibrous debris and generated fibrous debris are determined simultaneously.

5. Significance and Use

5.1 This test method can be used for acceptance testing of commercial shipments, but comparisons should be made with caution because information on estimates of between-laboratory precision is limited as noted in the precision and bias section of this test method.

5.1.1 If there are differences of practical significance between reported test results for two laboratories (or more), comparative tests should be performed to determine if there is a statistical bias between them, using competent statistical assistance. As a minimum, samples used for such comparative tests should be as homogeneous as possible, drawn from the same lot of material as the samples that resulted in disparate results during initial testing, and randomly assigned in equal numbers to each laboratory. Other fabrics with established test values may also be used for these comparative tests. The test results from the laboratories involved should be compared using a statistical test for unpaired data, at a probability level chosen prior to the testing series. If bias is found, either its cause must be found and corrected, or future test results must be adjusted in consideration of the known bias.

5.2 This test method rests on the hypothesis that a quantity of readily releasable fibrous debris (F_o [fibrous entities/m²]) is already present on the fabric, and that this debris is, by definition, easily removed therefrom merely by wetting the fabric with water and gently sluicing away the debris for subsequent enumeration. Generated fibrous debris (F_g [fibrous entities/m²]) is material which was either not initially present on the fabric or else was held very tightly within the matrix of the specimen.

5.3 Just as with particles,^{4,5} there is no unique answer for the quantity of fibrous debris that can be generated from such materials: the outcome depends on the kind and amount of energy administered. Obviously, a multiplicity of reasonable conditions exist under which fabrics can be made to generate fibrous debris, as well as a multiplicity of conditions under which this debris can be collected and enumerated. In this test method, an arbitrary but not unreasonable set of conditions are used. Because the results depend upon the volume of water

used in the shaking,⁶ a volume is chosen so that it is related to an inherent property of the fabric—namely, its sorptive capacity⁷—rather than employing an arbitrary single volume for all fabric specimens. The terms “fibrous debris,” “fibers,” and “fibrous entities” are used interchangeably throughout this test method.

5.4 This test method is useful to select fabrics with minimum release of fibrous debris during use. It can also be used to research fabrics for improved resistance to fibrous debris release and for production control.

6. Apparatus and Materials⁸

6.1 *Balance*, top loading, with a sensitivity of at least 0.01 g.

6.2 *Photographic Tray*, or equivalent, 300 mm × 500 mm × 60 mm (12 in. × 20 in. 2.4 in.).

6.3 *Beaker*, 2-L, (2-qt) capacity with 100 mL scale graduations.

6.4 *Jar*, poly(ethylene), or equivalent, 4-L (1-gal) capacity; height: 25 cm (10 in.); diameter: 15 cm (6 in.).

6.5 *Shaker*, having a frequency near 280 cycles/sec with amplitudes, respectively, of 17 mm and 8 mm, in the major and minor axes of the plane of oscillation, such as Tyler Model RX-86, or equivalent.

6.6 *Graduated Cylinders*, 10 mL, 25 mL and 500 mL.

6.7 *Filtration Apparatus*, for filtering suspensions of particles for subsequent enumeration, such as Millipore kit XX71 047-11, or equivalent.

6.8 *Membrane Filters*, black, 47.0-mm diameter, 0.8- μ m pore size, 3.0-mm grid squares, 100 squares per filtered membrane area.

6.9 *Microscope*, capable of resolving and sizing entities in range of interest, such as American Optical StereoZoom 7, or equivalent.

6.10 *Water*, at least distilled grade.

6.11 *Measuring Rule*, metal, graduated in 1 mm (0.05 in.).

6.12 *Die Cutter*, or equivalent, for 229 mm by 229 mm, \pm 1 mm (9.00 in. by 9.00 in., \pm 0.05 in.) specimens.

6.13 *Utility Knife*.

6.14 *Stirring Rod*.

6.15 *Hand Lens*, such as linen, pick, or magnifying glass having about 8 \times magnification.

7. Sampling and Test Specimens

7.1 *Primary Sampling Unit*—Consider rolls, bolts, or pre-packaged pieces of fabric to be the primary sampling unit, as applicable.

7.2 *Laboratory Sampling Unit*—As a laboratory sampling unit, use the primary sampling unit, as a source of test specimens and prepare the test specimens as directed in 7.3 and 7.4.

⁴ C. F. Mattina and S. J. Paley, “Assessing Wiping Materials for their Potential to Contribute Particles to Clean Environments: A Novel Approach,” *Particles in Gases and Liquids 2: Detection, Characterization and Control*, K. L. Mittal, Editor, 117-128, Plenum Publishing Corporation, New York (1990).

⁵ C. F. Mattina and S. J. Paley, “Assessing Wiping Materials for their Potential to Contribute Particles to Clean Environments: Constructing the Stress-Strain Curves,” *Journal of the IES*, 34(5), 21-28 (1991).

⁶ C. F. Mattina and J. M. Oathout, “Assessing Wiping Materials for their Propensity to Generate Particles: Biaxial Shaking Versus the Construction of Characteristic Curves,” *Proceedings, 40th Annual Meeting of the Institute of Environmental Sciences*, Chicago, Illinois, 1-6 May 1994, 20 (1994).

⁷ “Evaluating Wiping Materials Used in Cleanrooms and Other Controlled Environments,” IES-RP-CC004.2, Institute of Environmental Sciences, 940 East Northwest Highway, Mount Prospect, Illinois 60056, 1992.

⁸ Apparatus and materials are commercially available.

7.2.1 For primary sampling units having narrow widths or short lengths, use a sufficient number of pieces to prepare the test specimens described in 7.3 and 7.4.

7.3 *Test Specimen Size*—From each laboratory sampling unit, prepare three square test swatches, 229 mm by 229 mm (9.00 in. by 9.00 in.) from each laboratory sampling unit as directed in 7.4.

7.3.1 Primary sampling units may consist of prepackaged wiping material that are nominally 229 mm by 229 mm (9.00 in. by 9.00 in.) material squares. In those cases, use the entire square as the test specimen.

7.3.2 If prepackaged wiping material squares are folded, unfold them.

7.4 *Test Specimen Preparation*—Select test specimens as follows:

7.4.1 *For Prepackaged Wipes, Nominal 229 by 229 mm (9.00 in. by 9.00 in.)*—Open the package. Randomly select three wipes for test specimens. Use the entire square as the test specimen.

7.4.2 *For Rolls or Bolts of Fabric (Preferred)*—Using a utility knife, cut a plug, approximately 300 by 300 mm (12 by 12 in.) and deep enough into the roll or bolt to provide the necessary three fabric layers for test specimens. Using the die cutter, or equivalent, cut through the entire plug thereby providing the specimens necessary to meet the requirements of 7.3 and 7.4. (See Note 1 and A1.1.1).

7.4.2.1 Alternately, a full-width piece of fabric that is of sufficient length along the machine direction can be taken from the primary sampling unit to prepare the three test specimens after removing a first 1 m (1 yd) length. (See Note 1).

NOTE 1—Handle specimens with care and guard against contamination, abrasion or disturbing fibers that could contribute to an error in the fibrous debris count.

7.4.3 Take no specimens closer than 25 mm (1.0 in.) from the machine direction edge, except as noted in 7.3.1.

7.4.4 Ensure specimens are free of folds, creases, or wrinkles. Avoid getting oil, grease, etc. on the specimens when handling.

8. Conditioning

8.1 No conditioning is required unless otherwise specified in a material specification or contract order.

9. Preparation of Test Apparatus and Calibration

9.1 Verify that the balance is within calibration.

9.2 Verify that graduated cylinders, beakers and microscopes are within calibration.

10. Procedure

10.1 *Specimen Dimensions*—Measure and record the length (L) and width (W) of the specimen to the nearest 1 mm (0.05 in.).

10.2 *Extrinsic Sorptive Capacity*—Extrinsic sorptive capacity of the fabric to be tested must be known to determine generated fibrous debris. Establish as directed in 10.2.1 or 10.2.2, as applicable.

10.2.1 If the extrinsic sorptive capacity of a particular fabric is known, it is not necessary that extrinsic sorptive capacity be determined. Use known values, to meet the requirements of 10.4.2.

10.2.2 If extrinsic sorptive capacity is not known, determine it as directed in Test Method D 6651 as directed in Annex A1 and use to meet the requirements 10.4.2.

10.3 *Releasable Fibrous Debris (F_O)*—Determine releasable fibrous debris as follows:

10.3.1 Place a single ply test specimen flat in the center of the plastic tray.

10.3.2 Add approximately 500 mL of water to the tray so the specimen is completely covered.

10.3.2.1 The precise volume used is not critical and need not be known at this stage of the test.

10.3.3 Allow ample time (and/or use physical persuasion) so that the specimen sorbs as much liquid as possible, usually when no air bubbles are observed on the surface of the liquid.

10.3.4 After the specimen has sorbed water to its capacity, grasp the ends of the tray, lift it and alternating the tray ends in a smooth up-and-down motion, gently sluice the water across the specimen surface for 30 ± 3 s. Avoid delivering extraneous mechanical stress to the test specimen.

10.3.5 Decant the water into the 2-L (2-qt) beaker and reserve.

10.3.6 Using fresh water, repeat step 10.3.2-10.3.4 two additional times.

10.3.7 Measure and record the total volume (V_{OTB}) of water in the beaker to the nearest 50 mL.

10.3.8 Stir the suspension gently with a stirring rod, then aliquot it immediately using a graduated cylinder capable of reading the volume aliquotted to three significant figures. Record the aliquotted volume as (V_{OA}). (See Notes 2 and 3).

10.3.9 Using the filtration apparatus, filter the aliquotted suspension through the membrane filter.

NOTE 2—Unless a particular fabric is exceptionally low in fibrous debris, it will always be necessary to aliquot the suspension of fibrous debris for subsequent enumeration by optical microscopy. Because the proper volume cannot be known, a priori, sometimes more than one aliquot must be taken so as to deposit on the membrane filter a quantity of fibrous debris large enough to give sufficient statistical certainty, but not so large (higher than 25 entities per grid square) that the obscuration of some fibrous entities by others takes place. Fibrous debris of 5 to 25 entities for each grid is recommended. Generally, the aliquotted volume will be between 10 mL and 200 mL.

NOTE 3—It is sometimes helpful to examine the membrane filter with a hand lens immediately after the filtration of the aliquot in order to determine whether the volume aliquotted might possibly have been either too small or too large to provide the recommended number of fibrous entities per grid. In either case, the first aliquot can be replaced by a second aliquot using a different volume of aliquotted water taken immediately and using a fresh membrane. For some fabrics, the aliquotted volume may have an unusually high count of fibrous debris that makes counting difficult. When this occurs, dilute the aliquotted volume by a factor of 2 or more as needed to obtain counts less than 25 for each grid.

10.3.10 Air-dry the membrane filter in the test room atmosphere, shielded from dirt, lint or other air-borne particles.

10.3.11 Using a microscope with a calibrated eyepiece reticle, count and record the releasable fibrous debris ($C_{O1} + C_{O2} \dots C_{ON}$) on each grid of the filter membrane measured, and