



# Standard Specification for Polyolefin Monofilaments<sup>1</sup>

This standard is issued under the fixed designation D 3218; 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 specification covers polyolefin monofilament yarn materials, and test methods for standard polyolefin monofilaments. While designed primarily for testing standard polyolefin monofilaments, many of the procedures can be used, with little or no modification, for other polyolefin monofilaments. However, testing on non-standard polyolefin monofilaments should be conducted with caution. See 3.2.3 for a definition of standard polyolefin monofilament.

1.2 Only on condition that interlaboratory precision data are available for the specific procedure is any test method described, or referenced in this specification, recommended for acceptance testing of commercial shipments of polyolefin monofilaments.

1.3 The specification for polyolefin raw materials appears in Section 4.

1.4 The test methods for individual properties appear in the following sections:

Property	Section
Breaking Force	10
Breaking Tenacity	10
Elongation	10
Gloss	13
Hot Water Shrinkage	14
Initial Modulus	10
Polyolefin-Material Cleanliness	17
Resistance to Ultraviolet Radiation	15
Stability to Thermal Oxidation	16
Tensile Properties	10
Thickness	12
Width	11
Yarn Number	9

NOTE 1—In most instances, the suitability of these procedures for polymeric yarns in general, and polyolefin monofilaments in particular, is already accepted in commercial transactions (see 6.1).

1.5 The values stated in SI units are to be regarded as standard; the values in English units are provided as information only and are not exact equivalents.

1.6 The following safety hazards caveat pertains only to the test methods described in this specification: *This standard may involve hazardous materials, operations, and equipment. This*

*standard does not purport to address all of the safety problems, 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 76 Specification for Tensile Testing Machines for Textiles<sup>2</sup>
- D 123 Terminology Relating to Textiles<sup>2</sup>
- D 374 Test Methods for Thickness of Solid Electrical Insulation<sup>3</sup>
- D 1248 Specification for Polyethylene Plastics Molding and Extrusion Materials<sup>4</sup>
- D 1776 Practice for Conditioning and Testing Textiles<sup>2</sup>
- D 1907 Test Method for Linear Density of Yarn (Yarn Number) by the Skein Method<sup>2</sup>
- D 1921 Test Methods for Particle Size (Sieve Analysis) of Plastic Materials<sup>4</sup>
- D 2256 Test Method for Tensile Properties of Yarns by the Single-Strand Method<sup>2</sup>
- D 2258 Practice for Sampling Yarn for Testing<sup>2</sup>
- D 2259 Test Method for Shrinkage of Yarns<sup>2</sup>
- D 2565 Practice for Operating Xenon-Arc Type Light-Exposure Apparatus With and Without Water for Exposure of Plastics<sup>5</sup>
- D 4101 Specification for Propylene Plastic Injection and Extrusion Materials<sup>5</sup>
- E 203 Test Method for Water Using Karl Fischer Reagent<sup>6</sup>
- G 23 Practice for Operating Light-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials<sup>7</sup>
- G 26 Practice for Operating Light-Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials<sup>7</sup>

### 2.2 Other Documents:

- Federal Test Method Standard No. 141a, Sept. 1, 1965,

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<sup>2</sup> Annual Book of ASTM Standards, Vol 07.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 10.01.

<sup>4</sup> Annual Book of ASTM Standards, Vol 08.01.

<sup>5</sup> Annual Book of ASTM Standards, Vol 08.02.

<sup>6</sup> Annual Book of ASTM Standards, Vol 15.05.

<sup>7</sup> Annual Book of ASTM Standards, Vol 14.02.

Section 6000, Method 6101 “60-Degree Specular Gloss”<sup>8</sup>  
 Technical Report 24— “A Rapid Method for the Determination of Moisture in Pigmented Polyethylene Coating Materials,” Eastman Chemical Products Inc.<sup>9</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *breaking force*, *n*—the maximum force applied to a material carried to rupture.

3.1.2 *breaking tenacity*, *n*—the tenacity at the breaking force.

3.1.3 *draw ratio (DR)*, *n*—the relation of the final length per unit mass to original length per unit mass of a material resulting from drawing.

3.1.4 *drawing*, *n*—*in textile processing*, the process of stretching or attenuating a material to increase the length per unit mass.

3.1.4.1 *Discussion*—Drawing orients the molecular chains in the length direction of the monofilament.

3.1.5 *elongation at break*, *n*—the elongation corresponding to the breaking force.

3.1.5.1 *Discussion*—Elongation is expressed as a percentage of the length of the original specimen.

3.1.6 *gloss*, *n*—the luminous fractional reflectance of a material in the specular direction. (*Syn.* specular gloss)

3.1.7 *heat shrinkage*, *n*—a decrease in one or more dimensions of an object or material exposed to heat.

3.1.8 *initial modulus*, *n*—the slope of the initial straight portion of a stress-strain or force-elongation curve.

3.1.9 *monofilament*, *n*—a single filament which can function as a yarn in commercial textile operations, that is, it must be strong and flexible enough to be woven, knitted or braided, etc.

3.1.9.1 *Discussion*—(1) When a monofilament is drawn or in use, it does not fibrillate into (essentially) reticulate multifilaments. (2) A flat polyolefin monofilament can be called a tape yarn.

3.1.10 *polyolefin*, *n*—any long-chain synthetic polymer composed of at least 85 wt % of ethylene, propylene, or other olefin units (monomers), except amorphous (noncrystalline) polyolefin qualifying under Rubber 1, as defined by the Federal Trade Commission. (*Syn.* olefin)

3.1.10.1 *Discussion*—The generic term olefin has been adopted by the Federal Trade Commission in place of the technically correct term polyolefin.

3.1.11 *resistance to ultraviolet radiation*, *n*—the time to failure of yarns exposed to xenon-arc weathering.

3.1.11.1 *Discussion*—*In polyolefin tape yarns*, the failure criterion for resistance to ultraviolet radiation is the loss of 50 % of the original breaking tenacity.

3.1.12 For definitions of other textile terms used in this specification, refer to Terminology D 123.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *polyolefin-material cleanliness*, *n*—the degree to

which a polymer melt is free of filterable particles which remain insoluble in the melt under the specified test condition.

3.2.2 *stability to thermal oxidation*, *n*—*for polyolefin monofilaments*, the time-to-failure, when polyolefin monofilaments are exposed to circulating air at 125°C.

3.2.2.1 *Discussion*—The failure criterion for thermal oxidative stability is the mechanical breakdown described in Section 16.

3.2.3 *standard polyolefin monofilament*, *n*—*as used in this specification*, a flat polyolefin strand, approximately 0.05 mm (2 mils) thick by 2.5 mm (100 mils) wide and oriented with a draw ratio between 5:1 and 7:1.

3.2.3.1 *Discussion*—The strand is produced through slitting an extruded polyolefin film, and when drawn or in use does not fibrillate into (essentially) reticulate multifilaments.

3.2.4 *tape yarn*, *n*—yarn of a flat, tape-like character produced by slitting an extruded film.

## SPECIFICATIONS

### 4. Polyolefin-Monofilament Raw Materials

4.1 *Polyolefin Monofilaments* shall be made from either polypropylene as specified in 4.2, or polyethylene as specified in 4.3.

4.2 *Polypropylene* shall meet the requirements for Group 1 or 2, as detailed in Specification D 4101.

4.3 *Polyethylene* shall have a *density* higher than 940 kg/m<sup>3</sup> and shall meet the requirements for polyethylene plastics, as detailed in Specification D 1248.

4.4 *Flow Rate* of the polyolefin materials shall be agreed upon by the purchaser and the supplier, and shall be determined as directed in either Specification D 1248 or D 2146, whichever is applicable.

4.5 *Particle Size*—Shipments of polyolefin raw materials may be rated for particle size. When specified, particle size shall be determined by the multi-sieve analysis described in Method A of Test Methods D 1921.

4.6 *Polyolefin-Material Cleanliness*—Although resin cleanliness is not a structural or chemical characteristic, shipments may be advisable to rate shipments for the amount of foreign matter in, or on, delivered polyolefin raw materials.

4.6.1 When specified, polyolefin-material cleanliness shall be determined by the procedure described in Section 17 of this specification.

4.7 *Moisture Content*—Some monofilament-extrusion processes may be sensitive to slight amounts of moisture, inherently or otherwise present in the polyolefin raw material. In such cases, shipments may be rated for moisture content.

4.7.1 *Superficial Moisture Content* of polyolefin materials, when specified, shall be determined in accordance with the Procedure for Insoluble Solids in Test Method E 203.

4.7.2 *Total Moisture Content*, when specified, shall be determined in accordance with a method to be agreed upon

<sup>8</sup> Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

<sup>9</sup> Available from Eastman Chemical Products, Inc., Subsidiary of Eastman Kodak Co., P. O. Box 431, Kingsport, TN 37662.

between the purchaser and the supplier. The technique illustrated in Eastman Technical Report 24,<sup>10</sup> based on gas chromatography of vaporized moisture, is an acceptable analytical approach.

## TEST METHODS

### 5. Summary

5.1 Summaries of the various testing procedures are included in the referenced test methods, or in pertinent sections of this specification.

### 6. Significance and Use

6.1 *Acceptance Testing*—The test methods in Specification D 3218 for the determination of the properties of polyolefin monofilaments are considered satisfactory for acceptance testing of commercial shipments of polyolefin monofilaments, unless specified in the individual test method. These test methods are the best available and are used extensively in the trade.

6.1.1 If there are differences or practical significance between reported test results for two laboratories (or more) comparative test should be performed to determine if there is a statistical bias between them, using competent statistical assistance. As a minimum, test samples that are as homogeneous as possible, drawn from the material from which the disparate test results were obtained, and randomly assigned in equal numbers to each laboratory for testing. The test results from the two laboratories should be compared using a statistical test for unpaired data, at a probability level chosen prior to the testing series. If a bias is found either its cause must be found and corrected or future test results for that material must be adjusted in consideration of the known bias.

### 7. Sampling and Number of Specimens

7.1 Take samples as directed in the applicable material specification, or as agreed upon by the purchaser and the supplier. In the absence of an applicable material specification, or other agreement, take a lot sample and laboratory samples as directed in Practice D 2258.

NOTE 2—An adequate specification or other agreement between the purchaser and the supplier requires taking into account variability between shipping units, between packages, or ends within a shipping unit, and between specimens from a single package so as to provide a sampling plan with a meaningful producer's risk, consumer's risk, acceptable quality level, and limiting quality level.

7.2 The required number of specimens is covered in the referenced methods, or in the pertinent sections.

### 8. Conditioning

8.1 Expose the specimens in the standard atmosphere for testing textiles, as defined in Practice D 1776; except that it is not essential to control humidity.

### 9. Yarn Number

9.1 *Procedure*—Determine the direct yarn number in tex or

denier to three significant figures as directed in Option 1 of Test Method D 1907.

9.2 *Report*:

9.2.1 State that the specimens were tested as directed in Section 9 of Specification D 3218. Describe the material or product sampled and the method of sampling used.

9.2.2 Report the direct yarn number in tex, or in denier.

### 10. Tensile Properties

10.1 *Apparatus*—Tensile testing machine of a type as specified in Test Method D 2256. All types of tensile machines described in Test Method D 2256 are adequate to test polyolefin monofilaments with a draw ratio between 5:1 and 7:1. Polyolefin monofilaments with draw ratios outside this range cannot be tested with assurance of correct results, by all tensile machines specified in Test Method D 2256.

10.2 *Procedure*—Determine the breaking force, the breaking tenacity, and the elongation of adequately conditioned polyolefin monofilaments, using configuration A, condition 1 of Test Method D 2256.

10.3 *Report*:

10.3.1 State that the specimens were tested as directed in Section 10 of Specification D 3218. Describe the material or product sampled and the method of sampling used.

10.3.2 Report the following information for each laboratory sampling unit and for the lot:

10.3.2.1 Breaking force,

10.3.2.2 Breaking tenacity,

10.3.2.3 Elongation at break, as a percentage of the nominal gage length, and

10.3.2.4 Initial modulus.

### 11. Width

11.1 *Scope*—This test method covers the measurement of the width of polyolefin monofilaments, by means of a calibrated microscope.

11.2 *Summary of Test Method*—A specimen is placed on the microscope stage and is viewed under a magnification of 25×. The width of the specimen is measured using a reticle scaled eyepiece or filar micrometer eyepiece.

11.3 *Apparatus*:

11.3.1 Microscope designed for a magnification of 25×. With an eyepiece having a calibrated linear grid.

11.4 *Calibration of Apparatus*—Adjust the microscope, to secure the design magnification of 25×, and measure the total eyepiece scale using a stage micrometer, graduated in micrometers or mils. Calculate the conversion factor,  $F$ , to convert the eyepiece units to mils, using Eq 1:

$$F = M/N \quad (1)$$

where:

$M$  = stage micrometer readings, in micrometers (mils), and  
 $N$  = corresponding number of units in the eyepiece grid.

11.5 *Procedure*:

11.5.1 Adjust the microscope to the design magnification of 25×.

11.5.2 Place a specimen of the monofilament on the microscope stage, and set the scale of the eyepiece perpendicular to

<sup>10</sup> The Gardner Automatic Photometer Unit, Model AUX-3, available from Gardner Laboratory, Inc., P. O. Box 5728 (5221 Landy Lane), Bethesda, MD 20014, or its equivalent, has been found satisfactory for this method.

the long axis of the monofilament specimen.

11.5.3 Measure the width of the specimen monofilament, to the nearest eyepiece division. Repeat the width measurement three times, on different segments of the same specimen. Record the three width measurements.

11.5.4 Test four monofilament specimens.

11.6 *Calculation:*

11.6.1 Calculate the average width of the four specimens, in micrometers or mils, to three significant digits, using Eq 2:

$$\bar{X} = (\Sigma X)F / 12 \quad (2)$$

where:

$\bar{X}$  = average width of the four monofilaments,

$\Sigma X$  = sum of the twelve observed individual measurements, in eyepiece units, and

$F$  = conversion factor, as derived in 11.4.

11.7 *Report:*

11.7.1 State that the specimens were tested as directed in Section 11 of Specification D 3218. Describe the material or product sampled and the method of sampling used.

11.7.2 Report the average width of the four specimens, in micrometers or mils.

11.8 *Precision and Bias:*

11.8.1 *Precision*—The precision of this test method has not been established.

11.8.2 *Bias*—The procedure in Specification D 3218 for testing width has no known bias and is generally used as a reference method.

## 12. Thickness

12.1 *Scope*—This test method covers the determination of the thickness of flat polyolefin monofilaments, by a micrometer.

12.2 *Procedure:*

12.2.1 Determine the thickness of the monofilaments, as directed in Method C of Test Methods D 374. If it is necessary to test very narrow monofilaments, or round filaments, lay out several parallel specimens on the anvil.

12.2.2 Measure the thickness of the specimen to the nearest 2.5  $\mu\text{m}$  (0.1 mil). Repeat the thickness measurement three times on different segments of the same specimen. Record the three thickness measurements.

12.2.3 Make four tests for a total of 16 observations.

12.3 *Calculation:*

12.3.1 Calculate the average thickness of the four specimens, in  $\mu\text{m}$  (mils), to two significant figures.

12.4 *Report:*

12.4.1 State that the specimens were tested as directed in Section 12 of Specification D 3218. Describe the material or product sampled and the method of sampling used.

12.4.2 Report the average thickness of the four specimens.

12.5 *Precision and Bias*—The precision and bias of the procedures in Specification D 3218 for testing thickness are as specified in Test Methods D 374.

## 13. Gloss

13.1 *Summary of Test Method:*

13.1.1 Gloss is measured on both sides of a specimen

formed by winding three layers of the polyolefin monofilaments, in a standard pattern, on a yarn board. This procedure is especially designed for pigmented polyolefin monofilaments.

13.1.2 Basically, the method is derived from Federal Test Method Standard No. 141a, Method 6101.

13.2 *Significance and Use:*

13.2.1 The degree of gloss is important in many applications of polyolefin monofilament. This test method is used to provide a measure of this characteristic, from gloss readings on panels of polyolefin monofilaments wound on a yarn board.

13.2.2 Gloss readings are affected by many factors, such as: the degree of pigmentation of the specimen; the direction of the plane of the angle of incidence, relatively to the direction of the wind of the outer layer of monofilaments on the yarn board, when measuring unpigmented specimens with high gloss. When all these factors cannot be controlled, test results by this test method should be viewed with caution.

13.3 *Apparatus:*

13.3.1 *Gloss-Meter*<sup>10</sup>, graduated in 0.1-gloss units.

13.3.2 *Yarn Board Winder*—A small machine, usually operated by a hand crank, to rotate a yarn board end-over-end, and fitted with a traversing guide capable of spacing the yarn evenly across the board, as it is wound.

13.3.3 *Package Holders*—Vertical spindles for bobbins or cones, and shafts on which tubes or flanged spools can turn freely.

13.3.4 *Yarn Board*—Rectangle of stiff gray cardboard approximately 100 by 150 mm (4 by 6 in.).

13.4 *Procedure:*

13.4.1 Clamp one narrow end of the yarn board in the yarn board winder. The protruding end of the board in the clamp should extend about 100 mm (4 in.).

13.4.2 Lead the monofilament from the yarn package through the pigtail guide. Pass the first wrap of yarn around the width of the board and attach it to the upper right side of the yarn board. Push the pigtail guide to the extreme right on the traverse rod, and make sure the yarn is not twisted. Turn the yarn board slowly with the hand crank. After a few wraps, check the yarn on the board again, to see that it is not twisted. Wrap the yarn around the board neatly, so that each spiral of yarn lays immediately adjacent to the last previous wound spiral of yarn. Continue wrapping until the exposed area of the board is completely covered.

13.4.3 Remove the board from the winder, turn the board through a right angle and clamp the board, again, to the winder. Wrap a second layer of yarn at right angles to the first layer. Wrap the yarn across the board neatly, so that each spiral of yarn lays immediately adjacent to the next spiral of yarn. Cover as much as possible of the first layer.

13.4.4 Remove the board from the winder and turn the board to the position described in 13.4.1. Wrap the last layer of yarn (third layer) in the same manner described in 13.4.2 for the first layer.

13.4.5 Place the gloss-meter on the monofilament specimen (yarn board). Take six gloss readings at different places on one side, three longitudinally, and three transversely taking the readings directly from the instrument dial. Record the readings to the nearest 0.1-gloss unit.

13.4.6 Turn the board over and repeat 13.4.5.

13.5 *Calculation:*

13.5.1 Calculate the average of the 12 gloss readings for each of the laboratory sampling units to the nearest 0.1 unit and the average for the lot.

13.6 *Report:*

13.6.1 State that the specimens were tested as directed in Section 13 of Specification D 3218. Describe the material or product sampled and the method of sampling used.

13.6.2 Report the gloss for each laboratory sampling unit and for the lot.

13.7 *Precision and Bias:*

13.7.1 *Precision*—The precision of this test method has not been established.

13.7.2 *Bias*—No justifiable statement can be made on the bias of this method for determining gloss, since the true value of the property cannot be determined by an acceptable referee method.

## 14. Hot Water Shrinkage

14.1 *Procedure*—Determine the shrinkage in boiling distilled (or demineralized) water as directed in Test Method D 2259, except that the test specimens are single ends of polyolefin monofilament 1 m (39 in.) long rather than skeins, and the boiling time is 10 min. Measure the total length of each sample with a metre stick before and after shrinking. Apply a tension of 1 mN/tex (0.1 gf/tex) to remove any wrinkles that may be present. Test three specimens, and record the individual results.

14.2 *Report:*

14.2.1 State that the specimens were tested as directed in Section 14 of Specification D 3218. Describe the material or product sampled and the method of sampling used.

14.2.2 Report the average shrinkage of the three specimens, to the nearest 0.1 percentage point.

## 15. Resistance to Ultraviolet Radiation

15.1 *Summary of Test Method:*

15.1.1 The specimens are exposed to a xenon arc weatherometer source. The test conditions of temperature and humidity approximate those encountered in actual outdoors in subtropical regions.

15.1.2 Breaking tenacity is determined at regular intervals, during the laboratory exposure program and on an unexposed control yarn. The average breaking-tenacity values, obtained at each scheduled interval, are plotted versus the corresponding exposure times, in graph form. The graph is used to estimate (by interpolation) the exposure time corresponding to 50 % loss of original breaking tenacity.

15.2 *Significance and Use:*

15.2.1 Data obtained by this test method are expected to correlate reasonably well with data from exposure outdoors.

15.2.2 The key factor in this test method is the scheduling of the exposure interval, that is, the short-time exposure cycle within a total exposure program. After each exposure interval, the breaking tenacity is determined on an exposed specimen. Normally, five exposure intervals make up the exposure program.

15.2.3 For many applications of polyolefin monofilaments,

a 100-h exposure interval is adequate. Longer exposure intervals may be required.

15.3 *Preparation of Specimens:*

15.3.1 Mount at least four polyolefin monofilament segments on a specimen holder (the frame-type accessory supplied with each exposure apparatus), to make the mounted specimen. Use monofilaments long enough to be secured to both sides of the frame by all-weather plastic tape. Mount the filaments, fully extended but not stretched, 12.7 mm (0.5 in.) from each other, with no backing.

15.3.2 Prepare five mounted specimen holders.

15.4 *Apparatus*—As specified in Practice D 2565 or Practice G 26.

15.5 *Procedure:*

15.5.1 Expose five mounted specimens to xenon-arc weathering as directed in the recommended Procedure 4.1 of Practice D 2565.

15.5.2 At the end of each exposure interval, remove a mounted specimen holder from the apparatus and determine the breaking force of the four individual standard monofilaments, as directed in Section 10. Use a gage length of  $100 \pm 2$  mm ( $4.0 \pm 0.1$  in.). Also determine the breaking force of four unexposed control specimens. (Use the same length for the control as for the test.) Adjust the rate of operation to achieve a rate of strain equivalent to that used for testing in Section 10.

NOTE 3—An adequate specification or other agreement between the purchaser and the supplier requires taking into account variability between shipping units, between packages, or ends within a shipping unit, and between specimens from a single package so as to provide a sampling plan with a meaningful producer's risk, consumer's risk, acceptable quality level, and limiting quality level.

15.6 *Calculation:*

15.6.1 Calculate the breaking tenacity for the four specimens from each of the exposure intervals and for the unexposed control using the yarn number of the unexposed control.

15.6.2 Plot the average tenacities versus the corresponding times with zero time for the control. Estimate (by interpolation) the length of the exposure time, in hours, corresponding to 50 % loss of original breaking tenacity.

15.7 *Report:*

15.7.1 State that the specimens were tested as directed in Section 15 of Specification D 3218. Describe the material or product sampled and the method of sampling used.

15.7.2 Report the following information:

15.7.2.1 Resistance to radiative energy, as the estimated number of clock-hours, necessary to produce a 50 % loss of the original breaking tenacity, and

15.7.2.2 Artificial sunlight source, used in the exposure program.

15.8 *Precision and Bias:*

15.8.1 *Precision*—The precision of this test method has not been established.

15.8.2 *Bias*—No justifiable statement can be made on the bias of this method for determining resistance to ultraviolet radiation since the true value of the property cannot be determined by an acceptable referee method.

## 16. Stability to Thermal Oxidation

16.1 *Summary of Test Method*—Specimens of polyolefin

monofilaments are exposed to a current of air in a mechanical-convection oven at elevated temperature, until failure occurs.

#### 16.2 Significance and Use:

16.2.1 Under the severe conditions of this accelerated-aging test, the specimens undergo degradation at a rate that is dependent upon the thermal endurance of the polyolefin material. The time-to-failure by this test is a common criterion for assessing the stability to thermal-oxidation of polyolefin materials.

16.2.2 This test is not suitable for low melting olefins; that is, polyethylene and some olefin co-polymers.

#### 16.3 Apparatus:

16.3.1 *Oven*<sup>11</sup>—Mechanical-convection type for controlled circulation of air, with adjustable air intake and exhaust facilities, and designed for air velocities up to 1 m/s. It shall be equipped with a temperature-control system designed to maintain the oven temperature at  $125 \pm 1^\circ\text{C}$ . A device to prevent temperature overrides<sup>12</sup> shall be included in the temperature-control system.

16.3.2 *Thermometer*, with a temperature range from 0 to  $150^\circ\text{C}$ , graduated in intervals of  $1^\circ\text{C}$ .

16.3.3 *Glass Rod*, approximately 250 mm (6 in) long, with a diameter of approximately 12 mm (0.5 in).

#### 16.3.4 Air Meter.<sup>13</sup>

16.3.5 *Weights*—each with a 5-g mass in the form of wooden clothespins. Soft wire can be wrapped around one side of each pin to reach the required weight. Metal clips have been found unsatisfactory for this application.

#### 16.4 Preparation of Specimens:

16.4.1 Cut five polyolefin monofilament specimens, each approximately 250 mm (10 in.) long.

16.4.2 Secure the five specimens to the glass rod, by looping or tying one end. Maintain a 25-mm (1-in.) interval between specimens. Attach a 5-g weight to the free end of each specimen. The specimen assembly is now ready for mounting in the oven.

#### 16.5 Procedure:

16.5.1 Maintain the temperature at  $125 \pm 1^\circ\text{C}$  during thermal aging. To monitor this temperature, locate the thermometer bulb 75 to 80 mm from the top liner of the oven, in the vicinity of the exhaust port.

16.5.2 Maintain the nominal air velocity at  $0.5 \pm 0.1$  m/s, during thermal aging. Adjust the air intake and exhaust the ports, to allow at least one air-volume change per minute.

16.5.3 Suspend the specimen assembly from the upper shelf of the oven at the test temperature.

16.5.4 Inspect the specimens daily for failure, as evidenced by the material breakdown that causes a portion of the sample,

and its attached 5-g weight, to fall onto the bottom shelf of the oven.

16.5.5 Record the time-to-failure of each specimen, and calculate the average time-to-failure, in days, for the five specimens.

#### 16.6 Report:

16.6.1 State that the specimens were tested as directed in Section 16 of Specification D 3218. Describe the material or product sampled and the method of sampling used.

16.6.2 Report the stability to thermal oxidation as the average time-to-failure.

#### 16.7 Precision and Bias:

16.7.1 *Precision*—The precision of this test method has not been established.

16.7.2 *Bias*—No justifiable statement can be made on the bias of this test method for determining stability to thermal oxidation, since the true value of the property cannot be determined by an acceptable referee method.

## 17. Polyolefin—Material Cleanliness

17.1 *Scope*—This test method covers measurement of the cleanliness of molten polypropylene resins by filtration through a specified filter.

17.2 *Summary of Test Method*—Molten resin is extruded through an ultrafine screen filter. The rate of filter pressure increase measured during extrusion is an indication of melt resin cleanliness.

#### 17.3 Significance and Use:

17.3.1 This test method is particularly useful for product quality control tests on resins used in processes where the molten resin is filtered before extrusion. The melt cleanliness value determined by this method can be empirically related to filter life of processing equipment and, thereby, can be used to predict expected filter-life for a given lot of resin.

17.3.2 This test method is reproducible over a relatively wide range of cleanliness values and is suitable for use as a cleanliness specification for resins to be used in critical extrusion processes. It may be used for acceptance testing of commercial shipments because current estimates of between-laboratory precision are acceptable.

17.3.2.1 In cases of dispute, the statistical bias, if any, between the laboratory of the purchaser and the laboratory of the supplier should be determined with each comparison being based on testing randomized specimens from one sample of material.

17.3.3 The cleanliness value is based on filter pressure increase due to filter pluggage. It is assumed that no reaction occurs between resin or resin additives and the filter media and that resin melt viscosity remains constant during the filtration.

17.3.4 The filter pressure is influenced by changes in extrusion throughput or resin viscosity. It is important that extrusion rate be maintained at a constant level during a single filtration. Resin viscosity will change if processing temperature or resin thermal stability is nonuniform. Process temperature should be carefully maintained during each filtration. Temperature increases will reduce filter pressure and temperature decreases will increase filter pressure.

17.3.5 To compare the values for cleanliness from test to

<sup>11</sup> Freas Model 835, available from Precision Scientific Co., 3737 W. Cortland St., Chicago, IL 60647, or Blue M Model D6-560A, available from Blue M Electric Co., 138th and Chatham Sts., Blue Island, IL 60406, or an equivalent has been found satisfactory for this method.

<sup>12</sup> A bimetallic-strip temperature switch, Fenwal Thermoswitch, Catalog No. 1800-0, available from Fenwal Inc., 261 Main St., Ashland, MA 01721, or its equivalent, has been found satisfactory for this method.

<sup>13</sup> Anemotherm Model 60, available from Anemostat Product Division, Dynamics Corporation of America, P.O. Box 1083, Scranton, PA 18500, has been found satisfactory for this method.

test properly, the same weight of resin must be filtered each time.

17.4 Apparatus:

17.4.1 *Extruder*, designed to deliver a continuous supply of molten resin to the filter at a uniform rate, with good temperature control, through the operating pressure range. Typically, any 25 to 64-mm (1 to 2½-in.) commercial extruder is preferred for this test.

17.4.2 *Metering Pump* (optional)—Any positive displacement (gear type) metering pump commonly used for molten resins is satisfactory. The metering pump should have a capacity consistent with the extruder capacity and be capable of delivering a uniform volume of molten resin at the operating pressure range in use.

17.4.3 *Filter Assemblage*, composed of a series of stacked screens made, preferably, from Type 304 stainless steel, but Type 316 is satisfactory. Each filter screen has a support screen clipped to it as a pair with a stainless steel binding ring. All screens have a bound diameter of 56.64 to 56.77 mm (2.230 to 2.235 in.). Two types of paired screens are used with nominal pore sizes of 44 µm and 13 µm, respectively. Screen dimensions are shown in Fig. 1. The paired screens are stacked in the media holder in the following order: 44 µm (top), 44 µm, 13 µm, 44 µm, and 44 µm (bottom). In all cases, the 50 by 50 screen of each pair should be toward the bottom side of the holder.

17.4.3.1 The 44-µm screen has a top screen made from a plain weave 325 by 325 woven wire fabric using 0.0356-mm (0.0014-in.) diameter wire and having a hole width of 0.0432 mm (0.0017 in.). The bottom part of the 44-µm screen is made from a plain weave 50 by 50 woven wire fabric using 0.229-mm (0.009-in.) diameter wire and having a hole width of 0.279 mm (0.011 in.). The two screens are bound together with a retainer ring made from 0.254 mm (0.010 in.) stainless steel such that the width of the folded binder is no greater than 3.175

mm (0.125 in.) and the thickness of the binding rings is 1.35 to 1.40 mm (0.053 to 0.055 in.) as shown in Section AA of Fig. 1. The binding ring should be crimped sufficiently to secure the screens without crushing them, and the ring should be free of wrinkles. The completed screen assembly must be clean and free of metal clippings or foreign matter. The thickness must not vary more than 0.051 mm (0.002 in.) within each individual binding.

17.4.3.2 The 13-µm screen assembly has a top screen made from a Dutch twill weave 200 by 1200 wire fabric having diameters of 0.0660 mm (0.0026 in.) by 0.0406 mm (0.0016 in.). The bottom screen is identical to that used in the 44-µm screen assembly, and the two screens are bound under the same conditions and tolerances as the 44-µm screen assembly described previously.

17.4.3.3 The filter media holder has no critical outside dimensions except as needed to fit the extrusion equipment. It must have inside dimensions of 57.15 to 57.28-mm (2.250 to 2.555-in.) diameter and 31.75 to 31.88 mm (1.250 to 1.255 in.) deep. The bottom of the holder should contain 31 holes, 3.175 mm (0.125 in.) in diameter distributed in concentric circles (Fig. 2) with 4 holes located on a circle of 13.49-mm (0.531-in.) diameter, 9 holes located on a circle of 34.925-mm (1.375-in.) diameter and 18 holes located on a circle of 47.625-mm (1.875-in.) diameter. Outside dimensions of the holder are not critical, but the holder must be able to withstand the operating pressure range at 300°C.

17.4.4 *Pressure Gage*, covering the required range of filter pressures and having increments no larger than 0.689 MPa (100 psi) units. A system which continuously records pressure is preferred but not mandatory.

17.4.5 *Filtrate Collector*, capable of holding molten resin and allowing measurement of filtrate weight.

17.4.6 *Scale*, capable of accepting the filtrate collector and determining a net mass of filtrate up to 13.5 kg (30 lb) to the

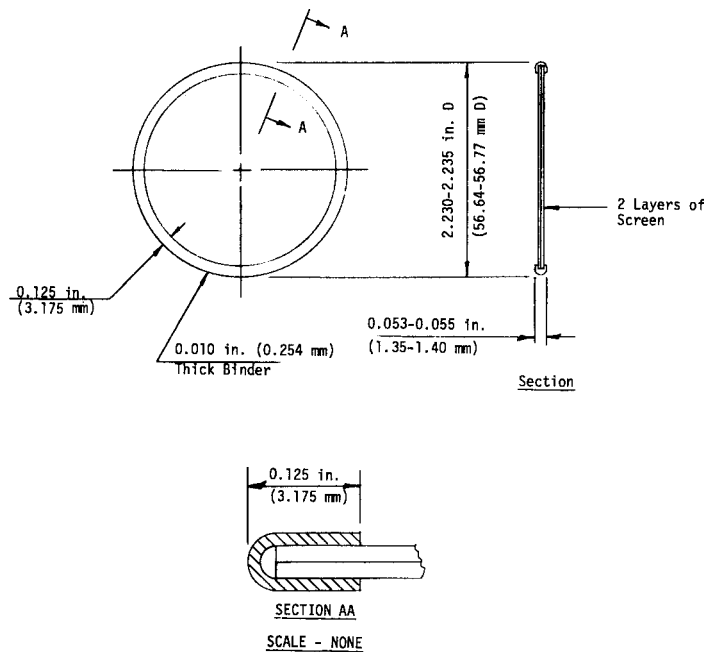
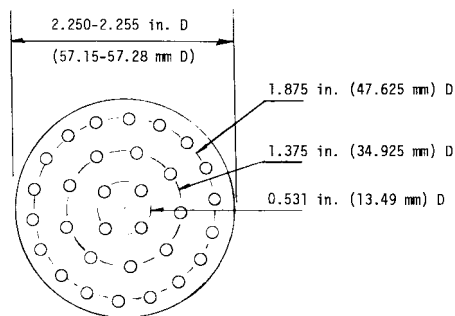


FIG. 1 Filtration Screen Details



**FIG. 2 Exit Hole Arrangement at Bottom of Filter Media Holder**

nearest 0.15 kg (0.3 lb).

**17.5 Test Specimen:**

17.5.1 The test specimen may be in any form that can be introduced into the throat of the extruder, preferably powder or granules. The form should be chosen such that the extruder can feed molten resin continuously without formation of voids in the melt.

17.5.2 Test one specimen from each laboratory sampling unit.

17.5.3 The test specimen size is dependent on the extrusion rate chosen for the test run. A size equal to or greater than 0.25 times the extrusion rate in kilograms (or pounds) per hour plus 13.6 kg (30 lb) must be used for the test. If the testing laboratory plans to run all filtrations at a chosen extrusion rate, then a standard test specimen can be designated for all test runs in that laboratory. In any event, the test specimen size should be such that after the purge is complete, a minimum of 13.6 kg (30 lb) of specimen remains for filtration.

**17.6 Procedural Conditions:**

17.6.1 Test conditions use a stock (resin) temperature of 265 ± 3°C. The extruder zone temperatures should be set as needed for the extruder in use to obtain the proper stock temperatures.

17.6.2 These conditions have been found satisfactory for polypropylene having a flow rate (FR) of 3 to 5 g/10 min. For resins having an FR outside of this range, a stock temperature should be chosen which is appropriate for the extrusion of the resin.

**17.7 Procedure:**

17.7.1 The equipment must be clean. A clean filter with only new (unused) screens must be used for each test.

17.7.2 Throughput level is not critical in this test. Any throughput consistent with safe operation of the equipment is acceptable. Since throughput influences the initial filter pressure, the maximum throughput allowable is one in which the initial pressure is at least 3.5 MPa (500 psi) below the safe operating pressure limit of the equipment in use or 34.5 MPa (5000 psi).

17.7.3 Assemble the filter by placing the paired screens in proper order in the holder and sealing them to prevent resin flow around the edges of the screen. The seal must not cover the exposed screen area. The filter must be fitted with a device to continuously measure pressure ahead of the filter. The filter can be assembled hot or cold. If assembled cold, it must be placed in an oven and heated up to stock temperatures before use (Note 4).

NOTE 4—It may be advisable to preheat the assembled filter to a

temperature of 10 to 15°C higher than stock temperature to avoid cooling below stock temperature during attachment of the filter assembly to the extruder. Attach the filter to the exit of the extruder, making sure that no leakage occurs ahead of the filter.

17.7.4 After the extruder has stabilized on set temperatures, charge the sample to the extruder feed hopper and purge the equipment for 15 min without the filter assembly in place at the extrusion rate chosen for the test. A minimum of 13.5 kg (30 lb) of sample must remain for filtration.

17.7.5 Stop extruder, attach filter assembly, begin filtration, and record, to the nearest 0.170 MPa (25 psi), the filter pressure when extrudate begins to exit the filter assembly (Note 5).

NOTE 5—At the start of the filtration, temperatures might not be at equilibrium after installing the filter. In this case, the initial pressure can be determined by extrapolating pressure back to zero time after the temperature has stabilized, providing no more than 10 % of the resin has been filtered during temperature stabilization.

17.7.6 Continue filtration (Note 6) monitoring the filter pressure until 13.6 kg (30 lb) of resin has been extruded (Note 7) or until back pressure reaches the safe operating limit or 34.5 MPa (5000 psi). Under no circumstances should the filter pressure decrease nor should the rate of increase become slower during the course of the test. When this occurs, discard the test result.

NOTE 6—After the filter is installed, the filter back pressure must be carefully watched as the extrusion is restarted. If a rapid pressure rise occurs, the extrusion must be stopped before the maximum safe operating pressures of the equipment are exceeded.

NOTE 7—The extrudate may be collected as a total mass and weighed after all resin has been extruded or an extrusion rate may be determined and the filtration continued for the time needed to filter 13.6 kg (30 lb) of resin.

17.7.7 Record, to the nearest 0.170 MPa (25 psi), the filter pressure at the point where 13.5 kg (30 lb) of resin has been collected. If the filter pressure reaches the safe operating limit or 34.5 MPa (5000 psi) before 13.5 kg (30 lb) of resin has been filtered, stop the test and weigh the resin filtered at that point.

17.7.8 Remove filter screens while hot and separate. Confirm the screens were of proper pore size (Note 8), placed in the holder in proper order (Note 9), and that no damage occurred to them during the filtration.

NOTE 8—It is important that all resin passes through the filter and that the critical screen maintains its pore size. Following the filtration, the filter should be examined to be sure that the screen stack has remained properly sealed at its circumference and that the screens have not been deformed (bulged). The test result should be discarded if either of these situations occur.

NOTE 9—The test reproducibility depends on the screens being stacked in proper order. Following each test, the screens should be removed and checked to see if properly stacked. If not, the test result should be discarded.

**17.8 Calculation:**

17.8.1 Calculate the pressure rise rate, for the material, in MPa/kg (psi/lb) using Eq 3:

$$\text{Rise Rate} = (P_f - P_i) / W \quad (3)$$

where:

$P_f$  = final filter pressure, MPa (psi),



$P_i$  = initial filter pressure, MPa (psi), and  
 $W$  = mass of resin extruded, kg (lb).

### 17.9 Report:

17.9.1 State that the specimens were tested as directed in Section 17 of ASTM Specification D 3218. Describe the material or product sampled and the method of sampling used.

17.9.2 The report shall include the following:

17.9.2.1 Stock temperatures, initial pressure, final pressure, and the mass of extrudate collected.

17.9.2.2 Resin cleanliness reported as filter pressure rise rate in pounds-force per square inch per pound (megapascals per kilogram) of extrudate filtered.

17.9.2.3 Melt flow rate of the resin.

17.9.2.4 Any unusual behavior of the equipment such as large temperature or extrusion rate variations.

17.9.2.5 Any unusual behavior of the test specimen or extrudate, such as discoloration, void formation, odor, particulate matter, or unusual viscosity.

### 17.10 Precision and Bias:

17.10.1 *Interlaboratory Test Data*<sup>14</sup>—An interlaboratory test was run in 1975 in which six laboratories each tested two specimens from each of two materials (a fiber grade and an injection-molding grade material). Each laboratory used one operator to test each material. The analyses were done using two independent runs on different days. All 12 specimens of each material came from the same sample. The components of variance expressed as standard deviations were calculated to be:

	Fiber Grade Materials
Within-laboratory component, psi/lb	1.23
Between-laboratory component, psi/lb	1.07

The results on the injection molding grade sample were erratic and did not allow statistical interpretation.

17.10.2 *Precision*—For the components of variance reported in 17.10.1, 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 1.

NOTE 10—Calculations were made using  $t = 1.960$ , which is based on infinite degrees of freedom.

<sup>14</sup> ASTM Research Report No. RR:D13-1052 is available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

**TABLE 1 Precision of Fiber Grade Materials**

Critical Difference, psi/lb for the Conditions Noted		
Number of Observations in Each Average	Within-Laboratory Precision	Between-Laboratory Precision
1	3.41	4.52
4	1.71	3.42
8	1.21	3.20

NOTE 11—This is 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.

17.10.3 *Bias*—No justifiable statement on bias can be made since the value of the property cannot be established by an accepted reference method.

## 18. Conformance

18.1 The purchaser and the supplier may agree on a procedure to establish conformance, including control charts furnished by the supplier, a sequential-sampling plan, or the double-sampling plan outlined in 18.2.

18.2 In the absence of a control-chart or sequential-sampling plan, proceed as directed in 18.2.1-18.2.3.

18.2.1 If the test results for the lot conform to the tolerances for all characteristics agreed upon, consider the lot a valid delivery.

18.2.2 If the test results for one or more characteristics do not conform to the tolerances, take a new laboratory sample from either the original lot sample or a new lot sample. Retest the lot for the characteristic(s) that did not conform to the tolerances in the first test, and average the results of the first and second samples as if all results were from one test of double the original number of specimens. If the new average(s) conform(s) to the specified tolerances, consider the lot a valid delivery.

18.2.3 If the test results obtained as directed in 18.2.2 do not conform to the specified tolerances, consider the lot a nonvalid delivery.

## 19. Keywords

19.1 appearance; chemical properties (textiles); dimensional change; impurity; polyolefin monofilaments; tension (tensile) properties/tests; thickness; width; yarn; yarn number

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