

Standard Specification for Ultra-High-Molecular-Weight Polyethylene Molding and Extrusion Materials¹

This standard is issued under the fixed designation D4020; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

- 1.1 This specification provides for the identification of virgin, natural color, unmodified homopolymer ultra-high-molecular-weight polyethylene (UHMW-PE) plastics molding and extrusion materials. This identification is made in such a manner that the seller and purchaser can agree on the acceptability of different commercial lots or shipments.
- 1.2 This specification also provides guidance for the characterization of UHMWPE materials based on various mechanical, thermal, electrical, and other analyses.
- 1.3 It is not intended to differentiate between various molecular weight grades of ultra-high-molecular-weight polyethylene commercially available.
- 1.4 It is not the function of this specification to provide specific engineering data for design purposes.
- 1.5 Ultra-high-molecular-weight polyethylenes, as defined in this specification, are those linear polymers of ethylene which have a relative viscosity of 1.44 or greater, in accordance with the test procedures described herein.
- 1.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.
- 1.7 The following precautionary caveat pertains only to the test method portions in Section 7 and the Annex and Appendixes, of this specification: 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.

Note 1—This standard and ISO 11542-1 address the same subject matter, but differ in technical content. ISO 11542-1 provides a classifica-

tion system based on various characteristics and a range of viscosity numbers determined in accordance with ISO 1628-3.

2. Referenced Documents

2.1 ASTM Standards:²

D883 Terminology Relating to Plastics

D1601 Test Method for Dilute Solution Viscosity of Ethylene Polymers

2.2 ISO Standards:³

ISO 11542-1 Plastics—Ultra High Molecular-Weight Polyethylene (PE-UHMW) Moulding and Extrusion Materials—Part 1: Designation System and Basis for Specification

ISO 1628-3 Plastics—Determination of Viscosity Number and Limiting Viscosity Number—Part 3: Polyethylenes and Polypropylenes

3. Terminology

- 3.1 *Definitions*—Definitions of terms used in this specification are in accordance with Terminology D883.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 ultra-high-molecular-weight polyethylene molding and extrusion materials—as defined by this specification, those substantially linear polyethylenes which have a relative viscosity of 1.44 or greater, at a concentration of 0.02 %, at 135°C, in decahydronaphthalene.
- 3.2.1.1 *Discussion*—It has been common practice to refer to the "molecular weight" of UHMW-PE resins. The following calculations shall be used to approximate the specific viscosity (η_{sp}), reduced viscosity (η red or R.S.V.), intrinsic viscosity (η or I.V.), and the approximate nominal viscosity average molecular weight of virgin resin. The calculations are shown as follows:

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

Relative viscosity =
$$\eta_r = \left(t_s - \frac{k}{t_s}\right) / \left(t_o - \frac{k}{t_o}\right)$$
 (1)

Specific viscosity =
$$\eta_{sp} = \eta_r - 1$$

Reduced viscosity =
$$\eta_{red} = \frac{\eta_{sp}}{C}$$

The intrinsic viscosity is calculated by determining the reduced viscosity and extrapolating to infinite dilution, that is, 0% concentration.

Intrinsic viscosity = [η] = $(2\eta sp - 2 \ln \eta rel)^{1/2} \div c$ Nominal viscosity molecular weight = $5.37 \times 10^4 [\eta]^{1.37}$

where:

k = kinetic energy correction constant for the particular viscometer used.

 $t_{\rm s}$ = flow time of solution at 135°C, s,

 $t_{\rm o}$ = flow time of pure solvent at 135°C, s, and

C =concentration.

Note 2—There are other equations being used in industry to calculate the nominal viscosity average molecular weights. Refer to Appendix X5 for the other equations and their relationship to the nominal viscosity average molecular weight equation in 3.2.1.1. The equation in 3.2.1.1 is the only equation that shall be used for reporting of nominal viscosity average molecular weight.

Note 3—Use of the solution viscosity test on thermally processed material is invalid due to inadequate solubility and possible crosslinking

4. Classification

4.1 It is recognized that dilute solution viscosity measurements can only be made on virgin resin. Therefore, the following test and limits shall be used to determine the properties of virgin polymer only.

5. Materials and Manufacture

- 5.1 The molding and extrusion material shall be UHMW polyethylene in the form of powder or granules.
- 5.2 The molding and extrusion materials shall be as uniform in composition and size and as free of contamination as can be achieved by good manufacturing practice. If necessary, the level of contamination shall be agreed upon between the seller and the purchaser.
- 5.3 Unless controlled by requirements specified elsewhere in this specification, the color and translucence of molded or extruded pieces, formed under conditions recommended by the manufacturer of the material, will be comparable within commercial match tolerances to the color and translucence of standard molded or extruded samples of the same thickness supplied in advance by the manufacturer of the material.

6. Sampling

- 6.1 A batch or lot shall be considered as a unit of manufacture and can consist of a blend of two or more production runs of the same material.
- 6.2 Unless otherwise agreed upon between the seller and the purchaser, prior to packaging, the material shall be sampled based on adequate statistical sampling.

7. Test Method

7.1 Dilute Solution Viscosity—Use Test Method D1601, as modified in Annex A1.

8. Keywords

8.1 extrusion materials; molding materials; plastics; polyethylene; ultra-high-molecular-weight; UHMW-PEviscosity

ANNEX

(Mandatory Information)

A1. DILUTE SOLUTION VISCOSITY

A1.1 General Description

A1.1.1 The test sequence consists of dissolving UHMW-PE in decahydronaphthalene (0.02 g/100 mL) at 150°C and then measuring the relative viscosity at 135°C in an Ubbelohde No. 1 viscometer. It is possible to calculate the relative solution viscosity from these experimental data.

A1.2. Apparatus

- A1.2.1 Analytical Balance.
- A1.2.2 Microscope Slide Cover Slip.
- A1.2.3 Hot Plate, with magnetic stirrer.
- A1.2.4 Erlenmeyer Flask, 250-mL, with glass stopper.
- A1.2.5 Vacuum Drying Oven.
- A1.2.6 Vacuum Aspirator.
- A1.2.7 Viscometer, Ubbelohde No. 1.

- A1.2.8 Constant-Temperature Bath, $135 \pm 0.1^{\circ}$ C, with a 305-mm diameter by 460 mm (12 by 18-in.) tall glass jar as a container, and having a suitable support for the viscometer.
 - A1.2.9 Buret, 100-mL capacity, 0.1-mL subdivisions.
 - A1.2.10 Stopwatch, 0.2-s reading.
 - A1.2.11 Still, for decahydronaphthalene.
 - A1.2.12 Glass Funnel, with heating mantle.

A1.3. Reagents

- A1.3.1 Decahydronaphthalene (Decalin), freshly distilled.
- A1.3.2 *Tetrakis* [methylene 3-(3',5'-di-*tert*-butyl-4'-hydroxyphenyl) propionate] methane (CAS No. 668-19-8).
- Note A1.1—This may also be referred to as Tetrakis-(methylene-(3,5-di-(tert)-butyl-4-hydrocinnamate))methane

A1.4. Procedure

A1.4.1 Stabilized Decahydronaphthalene Preparation—Distill in accordance with Test Method D1601 and add 0.2 % tetrakis [methylene 3-(3',5'-di-tert-butyl-4'-hydroxyphenyl) propionate] methane.

A1.4.2 Cleaning the Viscometer—Empty the viscometer thoroughly by vacuum and completely refill the viscometer with distilled, filtered, non-stabilized decahydronaphthalene. Place the viscometer into the 135°C hot oil constant temperature bath for at least 15-20 min. Completely drain the viscometer and dry with dry air or nitrogen just prior to the next measurement in order to prevent dilution and an erroneous measurement result.

A1.4.3 Solution Preparation—Dry the UHMW-PE in a vacuum oven for 2 h at 60°C. Weigh 14 to 17 mg of the dry UHMW-PE onto a slide cover slip. Use the buret to transfer the stabilized decahydronaphthalene at room temperature into the Erlenmeyer flask, measuring, in millilitres, a volume equal to 4.5 times the UHMW-PE weight in milligrams, for example, 15 mg of UHMW-PE and 67.5 mL of decahydronaphthalene. Heat the decahydronaphthalene, with stirring, to 150°C, and drop in the UHMW-PE and its slide cover slip. Continue stirring at 150°C for 1 h, with the flask lightly stoppered.

A1.4.4 Viscosity Measurement:

A1.4.4.1 Place the clean viscometer into the constant-temperature bath, fill with stabilized decahydronaphthalene, and allow the viscometer and solvent to come to thermal equilibrium at 135 ± 0.1 °C. Determine the viscosity of the solvent. Clean the viscometer as directed in A1.4.2. It is essential that the whole viscometer be dry.

A1.4.4.2 Meanwhile, place the flask of polymer solution into the 135°C bath and allow it to equilibrate. Transfer sufficient solution to fill the viscometer to the mark (see Note A1.2) and determine the viscosity of the solution.

A1.4.4.3 Between uses, clean the viscometer as described in A1.4.2. Prolonged waits between uses (overnight, etc.) will require the use of the $H_2SO_4 - K_2Cr_2O_7$ cleaning solution.

Note A1.2—Filling of the viscometer is made easier by the use of a glass funnel warmed with a heating mantle. This helps to prevent the UHMW-PE from precipitating.

A1.5. Calculation

A1.5.1 Calculate the relative solution viscosity as follows:

$$\eta_r = (t_s - k/t_s)/(t_0 - k/t_0) \tag{A1.1}$$

where:

k = kinetic energy correction constant for the particular viscometer used,

 $t_{\rm s}$ = flow time of solution at 135°C, and

 t_0 = flow time of pure solvent at 135°C.

APPENDIXES

(Nonmandatory Information)

X1. CHARACTERIZATION OF ULTRA-HIGH-MOLECULAR-WEIGHT POLYETHYLENE

X1.1 Scope

X1.1.1 The following appendixes provide guidance for the characterization of UHMW-PE based on various mechanical, thermal, electrical, and other analyses.

X2. IMPACT TEST METHOD FOR ULTRA-HIGH-MOLECULAR-WEIGHT POLYETHYLENE

X2.1. Scope

X2.1.1 This test method covers determination of the impact strength of UHMW-PE, which is extremely impact resistant. When tested in accordance with Test Method D256, Method A, UHMW-PE generally gives the NBF type of failure, rendering the test result invalid. This test method specifies the same type of pendulum impact test machine as that given in Test Method D256 but introduces a much higher degree of stress concentration into the specimen by double notching with a razor blade. Application of this test method shall be limited to the characterization of virgin, unmodified UHMW-PE resins, not commercially processed products. It is advised that the user be familiar with Test Method D256 before attempting to use this test method.

X2.1.2 The values stated in SI units are to be regarded as the standard.

Note X2.1—This test method and Annex B of ISO 11542-2 address the same subject matter, but differ in technical content and results shall not be compared between the two test methods.

X2.2. Referenced Documents

X2.2.1 ASTM Standards:²

D256 Test Methods for Determining the Izod Pendulum Impact Resistance of Plastics

X2.2.2 ISO Standards:³

ISO 180-1982 (E) Determination of Izod Impact Strength of Rigid Materials

ISO 11542-2 Plastics—Ultra-High Molecular Weight Polyethylene (PE-UHMW) Moulding and Extrusion



Materials—Part 2: Preparation of Test Specimens and Determination of Properties

X2.3. Apparatus

X2.3.1 The Izod-type impact machine that conforms to the requirements of Test Method D256, including the calibration and checking methods, shall be used.

X2.4. Test Specimen

X2.4.1 The geometry and dimensions of the specimen are given in Fig. X2.1.

X2.4.2 The specimens shall be cut from a sheet compression molded in accordance with the conditions described in Table X2.1:

TABLE X2.1 Molding Conditions for UHMW-PE Impact Test Specimens

Molding pressure	6.9 to 10.3 MPa
Platen temperature	196 to 210°C
Heating time	20 min at 196 to 210°C
Platen cooling rate	15 ± 2°C/min from 150 to 90°C
Platen temperature for demolding	<30°C

X2.4.3 The width of the specimen shall be the thickness of the sheet if the sheet thickness is within 6.00 to 6.75 mm. Sheet material thicker than 6.75 mm shall be machined down to 6.35 \pm 0.25 mm. Sheet material thicker than 7.65 mm shall not be used.

X2.4.4 Each specimen shall be free of twist and shall be bounded by mutually perpendicular pairs of plane parallel surfaces, free from scratches, pits, and sink marks.

X2.5 Notching of Specimens

X2.5.1 Notching shall be performed on the side parallel to the direction of the application of molding pressure.

X2.5.2 Notching shall be performed in a suitable machine by pressing in a 0.23 ± 0.03 -mm thick single-edge razor blade with a $14 \pm 2^{\circ}$ included angle at the cutting edge. The notching speed shall be less than 500 mm/min. A new blade shall be used after notching 40 specimens.

X2.5.3 The calibration of the notching machine shall be checked by direct measurement of the notch depth,

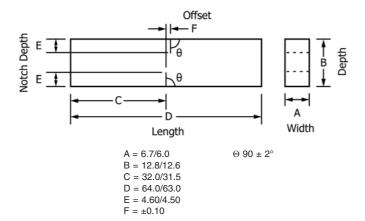


FIG. X2.1 Dimensions of Double-Notched Izod Test Specimens

perpendicularity, and offset of the two notches. One of the possible measurement methods is given in Appendix X3.

X2.6. Conditioning

X2.6.1 *Conditioning*—Condition the notched specimens at $23 \pm 2^{\circ}$ C for not less than 40 h prior to test.

X2.6.2 *Test Conditions*—Conduct the test in the standard laboratory atmosphere of $23 \pm 2^{\circ}$ C.

X2.7. Procedure

X2.7.1 At least five and preferably ten individual determinations of impact value must be made on each sample to be tested under the conditions prescribed in X2.6.

X2.7.2 Measure the width of each specimen in the region of the notches twice with a micrometer to the nearest 0.025 mm, and record its average width. Use an optical microscope to measure the distances between the notch roots on the two side surfaces of the specimen. Record the average value and multiply this number by the width of the specimen to obtain the remaining unnotched cross-section area, *AR*. Also record the identifying markings of the specimen.

X2.7.3 Estimate the breaking energy for the specimen and select a pendulum of suitable energy. Start the test with a pendulum of 11 J if no prior test data are available. Use the lightest standard pendulum that is expected to break each specimen in the group with a loss of not more than 85 % of its energy.

X2.7.4 Before testing the specimens, perform the following operations on the machine:

X2.7.4.1 With the excess energy indicating pointer in its normal starting position, but without a specimen in the vise, release the pendulum from its normal starting position and note the position that the pointer attains after the swing as one reading of Factor A.

X2.7.4.2 Without resetting the pointer, raise the pendulum and release again, which will move the pointer up the scale an additional amount. Repeat this step if the pointer does not move. Repeat this procedure until a swing causes no additional movement of the pointer, and note the final reading as one reading of Factor B.

X2.7.4.3 Repeat the above two operations several times, and calculate and record the average A and B readings.

X2.7.5 Position the specimen precisely and rigidly but not clamped too tightly in the vise. The relationship of the vise, specimen, and striking edge of the pendulum to one another is given in Fig. X2.2. Note that the top plane of the vise shall be 0.13 ± 0.13 mm below the notches.

X2.7.6 Release the pendulum and note and record the excess energy remaining in the pendulum after breaking the specimen.

X2.7.7 From the breaking strength of the specimen and Factors *A* and *B*, determine the energy loss of the pendulum due to windage and friction using the correction charts from the commercial testing machine supplier. If these charts are not available, use the method given in Appendix X2 or X3 of Test Method D256. Subtract the correction so calculated from the

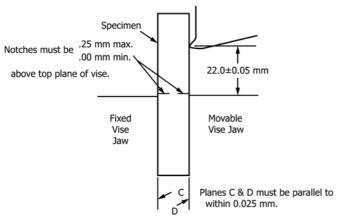


FIG. X2.2 Relationship of Vise, Specimen, and Strike Edge to One Another

indicated breaking strength of the specimen. If a pendulum of improper energy was used, discard the result and make additional tests on new specimens with the proper pendulum. If the proper pendulum was used, divide the net value so found by the unnotched area AR of the specimen as measured in X2.7.2 to obtain its impact strength in kilojoules per square metre.

- X2.7.8 Record the type of failure for each specimen as one of the two coded categories defined as follows:
- (1) C, Complete Break—A break in which the specimen separates into two pieces.
- (2) NB, Non-Break—A break in which the specimen does not separate into two pieces.
- X2.7.9 Calculate the average impact strength and standard deviation of the group of specimens that results in complete breakage. This test method requires that the specimen breaks completely. The results obtained from unbroken specimens shall be considered a departure from standard and shall not be reported as a standard result.

X2.8. Report

- X2.8.1 Report the following information:
- X2.8.2 Complete identification of the material tested, including type, source, manufacturer's lot number, and previous history;

- X2.8.3 Compression molding conditions;
- X2.8.4 Capacity of the pendulum, J;
- X2.8.5 Total number of specimens tested;
- X2.8.6 Number of those specimens that result in complete break;
 - X2.8.7 Average impact strength, kJ/m²;
 - X2.8.8 Standard deviation; and
- X2.8.9 Percent of specimens failing in each category, suffixed by the corresponding letter code from X2.7.8.

X2.9. Precision and Bias

X2.9.1 Table X2.2 is based on a round robin conducted by seven laboratories. For each material, all of the test specimens were compression molded and machined at one source. Each participating laboratory notched and tested five specimens of each material.

X2.9.1.1 Repeatability, I_r (Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results are judged not equivalent if they differ by more than the I_r value for that material.

X2.9.1.2 Reproducibility, I_R (Comparing two test results for the same material, obtained by different operators using different equipment on different days)—The two test results are judged not equivalent if they differ by more than the I_R value for that material.

TABLE X2.2 Precision of the Double-Notched Izod Impact Test
Method

Material	Intrinsic Viscosity,		١	/alues, kJ/m	1 ²	
iviateriai	dl/g	Mean	S_r^A	$S_R^{\ B}$	I_r^C	I_R^D
А	24	128.0	6.5	27.6	18.4	78.2
В	27	120.0	5.4	25.8	15.2	73.1
С	22	103.9	4.1	21.2	11.6	59.9
D	28	56.1	2.2	9.6	6.2	27.2
Е	25	63.5	2.7	12.6	7.7	35.5

 $^{{}^{}A}S_{r}$ = within-laboratory standard deviation of the average.

 $^{{}^{}B}S_{R}$ = between-laboratories standard deviation of the average

 $^{^{}C}I_{r} = 2.83 S_{r}$

 $^{^{}D}I_{R} = 2.83 \ S_{R}$

X3. MEASUREMENT METHOD OF IMPERFECTIONS IN SPECIMEN NOTCHING

X3.1 The following is one of the possible test methods for measuring the imperfections in specimen notching directly, which can be classified into three kinds: (1) deviation from perpendicularity, (2) incorrect notch-depth, and (3) offset of notches (Fig. X3.1).

Note X3.1—There is no known ISO equivalent to this method.

X3.2 Apparatus

X3.2.1 Reflective Optical Microscope, ocular, 40 to 60x, with an X-Y stage accurate to 0.0025 mm.

X3.2.2 Eyepiece, with a crosshair.

X3.2.3 Fiber Optic Illumination.

X3.3 Procedure

X3.3.1 Lay the specimen on one of its sides and mount it securely on the *X-Y* stage.

X3.3.2 The beginning and ending points of the notches are labeled from *A* to *D* in Fig. X3.1. Select one of the edges of the specimen as the datum line from which the perpendicularity of

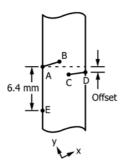


FIG. X3.1 Notch Geometry of Double-Notched Izod Specimen

the notches to the edges is measured (in this case Line $A\overline{E}$). Note that Point *E* is approximately 6.4 mm from Point *A*.

X3.3.3 Keep both the microscope and the base of the X-Y stage stationary. Measure the coordinates of Points A to E with respect to an arbitrarily selected coordinate system by moving the X-Y stage and by targeting the points by the crosshair of the eyepiece.

X3.4 Calculation

X3.4.1 The following equation is used to calculate the perpendicularity of the notches:

$$\angle EAB = \tan^{-1} \frac{m_2 - m_1}{1 + m_2 m_1}$$
 (X3.1)

where:

 m_1 and m_2 = slopes of line \overline{AE} and \overline{AB} with respect to the coordinate system.

 m_1 and m_2 are calculated from

$$m = \frac{y_2 - y_1}{x_2 - x_1} \tag{X3.2}$$

where:

m = slope, and (x_1, y_1) and (x_2, y_2) = coordinates of the end points of the line.

The distance between two points, I, is obtained from the following equation:

$$I = \sqrt{(x_2 - x_1)^2 + (y_2 - y_1)^2}$$

The amount of offset of the notches is calculated from the following equation:

offset =
$$|AD| \cos \angle DAE$$
 (X3.4)

X4. ELONGATIONAL STRESS TEST METHOD FOR ULTRA-HIGH MOLECULAR-WEIGHT POLYETHYLENE

X4.1. Scope

X4.1.1 This test method covers the determination of elongational stress as a characterization of the melt viscosity of UHMW-PE. The melt flow rate in accordance with test method D1238 cannot be determined for this material because ultra high molecular weight polyethylene does not have a melt flow. The elongational stress is also be referred to as ZST and flow value, or both.

X4.1.2 Application of this test method shall be limited to virgin, unmodified resin. The elongational stress method is invalid on a previous thermally-processed material due to possible crosslinking.

Note X4.1—This test method is identical to to Annex A of ISO 11542-2 in the measurement of elongational stress. It is not equivalent to ISO 11542-2 in any other measurement or section

X4.2. Referenced Documents

X4.2.1 ASTM Standards:²

D4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets

X4.2.2 ISO Standards:

ISO 11542–2 Plastics – Ultra-high-molecular-weight polyethylene (PE-UHMW) Moulding and Extrusion – Part 2:
 Preparation of test specimens and determination of properties

X4.3. Terminology

X4.3.1 *elongational stress*—(in MPa) the tensile stress (force related to the initial cross-sectional area) required to elongate a test specimen 600 % in a hot oil bath at 150°C in a 10-min time period.

X4.3.2 *tensile stress*—(in MPa) the attached weight corrected for the buoyancy effect divided by the measured initial cross-sectional area.

X4.4. Apparatus

X4.4.1 Specimen die cutter

X4.4.2 Specimen holder in accordance with Fig. X4.1

X4.4.3 Constant temperature bath with thermoregulator and circulating pump

X4.4.4 Graduated weight set with hooks for suspension from the specimen holder. Recommended weights are approximately 700, 650, 600, 550, 500, 450, 400, 350, 300, 250, 200, 180, 150, 120, and 100 g.

X4.4.5 Measuring instrument capable of measuring to 0.02 mm

X4.4.6 Stopwatch

X4.4.7 Hot bath liquid (for example, silicone oil)

X4.4.8 Compression molding press with controlled rate of cooling of 15 ± 2 °C/min

X4.4.9 Positive compression mold with a minimum of 4 grooves for venting and minimization of residual stress and warpage. Capable of molding plaque or disk 1.4 mm in thickness.

X4.4.10 Aluminum foil

X4.4.11 Analytical balance, accurate to ± 0.1 g

X4.4.12 Blender, high intensity

X4.5. Reagents

X4.5.1 The addition of a mixture of a primary and secondary antioxidant to reduce the amount of crosslinking taking place in the specimens. The type and amount of antioxidant used will depend on the lab and the R^2 value observed when results are calculated.

Note X4.2—A blend of Tris(2,4-di-(tert)butylphenyl)phosphite (CAS No. 31570-0404) and Tetrakis [methylene 3-(3',5'-di-tert-butyl-4'-hydroxyphenyl) propionate] methane (CAS No. 6683-19-8) at a 2:1 or 1:1 ratio has been found to work well when added at between 0.4 and 0.75 % by weight.

X4.6. Procedure

X4.6.1 Test Plaque Preparation:

X4.6.1.1 Using the analytical balance weigh out the amount of UHMW-PE virgin material that will be needed to mold the number of plaques or disks required for the study. Based on the amount of UHMW-PE weighed, weigh the amount of antioxidant necessary to achieve a concentration capable of reducing

crosslinking. With the high intensity blender, mix the antioxidant homogeneously into the UHMW-PE.

X4.6.1.2 Place the bottom half of the positive mold on a flat surface. Cover the bottom half with a piece of aluminum foil. Weigh out the amount of the UHMW-PE/antioxidant mix necessary to fill the mold, make a full part, and minimize flash and warpage. When this weight is established, this weight $\pm 0.1~g$ shall be used consistently to ensure uniform moldings. Pour the weighed polymer into the mold cavity and spread it out into a smooth level surface. Cover with a second piece of aluminum foil, then the upper portion of the positive compression mold.

X4.6.1.3 A completely fused test plaque is prepared by compression molding. The following molding conditions are proposed as guidelines: 1) 200°C under 12.7 MPa pressure for 20 min, 2) cool under pressure at a cooling rate of 15 \pm 2°C/min and 3) when the plaque or disk has cooled to below 40°C, remove it from the mold.

X4.6.2 *Test Specimens*—Six specimens in accordance with Fig. X4.2 shall be die cut out of one test plaque. Each specimen is tested using a different weight as described in X4.6.4.4.

X4.6.3 Measurement of Cross-sections—The width and thickness at the narrow parallel-sided section of each of the six specimens shall be measured and recorded to the nearest 0.02 mm

X4.6.4 Elongational Stress Determination

X4.6.4.1 Stabilize the bath at 150 \pm 2°C.

X4.6.4.2 Insert test specimen in the holder, hook the corresponding weight to the holder and suspend in bath. The mass of the holder and weights shall be known to an accuracy of 0.1 g.

X4.6.4.3 After preheating five min, elongate specimen and record specimen elongation time. The elongation of test specimens does not take place at constant speed.

X4.6.4.4 Repeat for the remaining specimens. The choice of the six different weights used to load test specimens from the weights listed in X4.4.4 depends upon the molecular weight of the UHMW-PE sample. The weights shall be selected so that a time of 1 to 20 min gives 600 % elongation in the narrow parallel-sided section of the test specimen.

X4.7. Calculation

X4.7.1 The tensile stress in MPa on each individual specimen is calculated in accordance with the following equation:

$$T_s = \left\{ \left[\left(M_{\scriptscriptstyle 1} + M_{\scriptscriptstyle 2} \right) \times 0.00981 \right] / A_{\scriptscriptstyle 1} \times B_{\scriptscriptstyle 1} \right\} \times \left[1 - \left(\rho_{\scriptscriptstyle m} / \rho_{\scriptscriptstyle w} \right) \right] (\mathrm{X4.1})$$

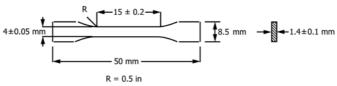


FIG. X4.1 Test Specimen



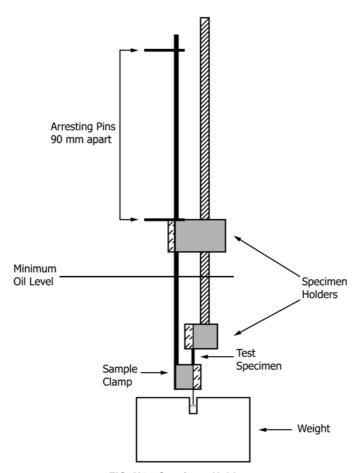


FIG. X4.2 Specimen Holder

 T_S = Tensile stress in MPa M_I = Mass of selected weight M_2 = Mass of specimen holder

= Initial thickness of test specimen (mm) = Initial width of test specimen (mm)

= Density of the heat bath medium at 150°C

= Density of the metal at 150°C

Use log/log scale to plot tensile stress for the six specimens against corresponding times for the 600 % elongation recorded in X4.6.4.4. Draw a best fit line through the six points and from this graph, read off the tensile stress corresponding to a period of 10 min. This value represents the elongational stress in MPa.

Note X4.3—An undue amount of scatter ($R^2 \le 0.95$) or zero elongation indicates that crosslinking has occurred in the test specimens. If this occurs, repeat the test using specimens prepared using an increased amount of stabilizer. Increase the stabilizer level at least 50 % from what was previously used.

X4.8. Precision

X4.8.1 The repeatability standard deviation has been determined to be 0.009 MPa for a material having an average elongational stress value of 0.516 MPa. This is based on a single laboratory making 45 measurements over a period of time on one material. The reproducibility of this test method is not yet available.



X5. NOMINAL VISCOSITY AVERAGE MOLECULAR WEIGHT OF ULTRA-HIGH MOLECULAR-WEIGHT POLYETHYLENE

X5.1. Scope

X5.1.1 The measurement of molecular weight of UHMW-PE is nearly impossible to measure by techniques normally used to measure molecular weight of other polymers. Techniques such as gel permeation chromatography (GPC) and light scattering used for other polymers are not useful for UHMW-PE.

X5.1.2 The dilute viscosity test method can provide satisfactory correlations for viscosity average molecular weight within a specific manufacturing process, but this does not

necessarily apply for another manufacturing process. As a result, at least five equations have been developed to describe the molecular weight of UHMW-PE.

X5.1.3 Fig. X5.1 shows the relationship between the five equations with respect to nominal viscosity average molecular weight versus intrinsic viscosity.

X5.1.4 This appendix is being provided only as a reference. Only Eq 1 listed in 3.2.1.1 of this specification shall be used to present data to the industry.

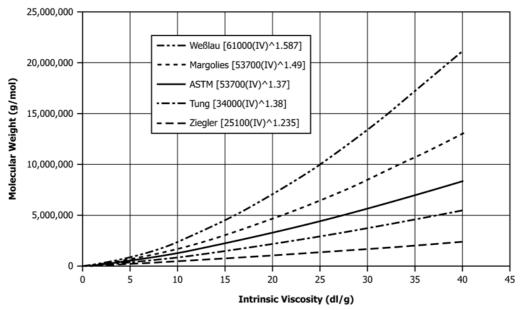


FIG. X5.1 Known Molecular Weight Equations (Correlating with Intrinsic Viscosity)

X6. ABRASION INDEX OF UHMWPE USING AN ALUMINUM OXIDE SLURRY

X6.1. Scope

X6.1.1 This method is used to determine the resistance of materials to abrasion, measured in terms of percent weight loss, by rotating test specimens in a slurry consisting of 20 grit aluminum oxide and water.

X6.1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

Note X6.1—There is no known ISO equivalent to this method.

X6.2. Referenced Documents

X6.2.1 ASTM Standards:²

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D1921 Test Methods for Particle Size (Sieve Analysis) of Plastic Materials

D4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

X6.3 Terminology

X6.3.1 *Definitions*—For definitions related to plastics, see Terminology D883.

X6.4. Apparatus

X6.4.1 Abrasion tester, consisting of:

X6.4.1.1 *Motor*—capable of maintaining a uniform rotational speed of 1750 rpm.

X6.4.1.2 Stainless steel testing cups—140 mm (5.5 in.) deep and 102 mm (4 in.) inside diameter, for holding specimens and aluminum oxide slurry.

X6.4.1.3 Stainless steel (SS) shafts—9 mm outside diameter.

X6.4.1.4 Metal cup supports—with dimensions of 3.2 mm by 51 mm by 152 mm (1/8 in. by 2 in. by 6 in.)

X6.4.1.5 *Thermocouple*—positioned in the testing cup to monitor slurry temperature.

X6.4.1.6 *Gaskets*—fitted into groove at the top of each testing cup to prevent leaking of slurry during motor rotation.

X6.4.1.7 Water bath—plastic glass container having dimensions of 55 by 53 by 20 cm. Contains the water to cool the testing cups during operation.

X6.4.1.8 *Hydraulic jack*—for raising the water bath up under the testing cups.

X6.4.1.9 *Control panel*—consisting of a timing device for automatic test termination after 2 h and other controls.

X6.4.1.10 *Water chiller*—to maintain the water bath at $23 \pm 2^{\circ}$ C.

X6.4.1.11 *Thermocouple*—to verify the temperature of the water bath

X6.4.2 *Analytical balance*—for weighing test specimens, accurate to 0.0001 g.

X6.4.3 *Pan balance*—with approximately a 1000 g capacity for preparation of slurry.

X6.4.4 *Vented rack*—for air-drying specimens, to allow for free circulation of air.

X6.5. Reagents

X6.5.1 Aluminum Oxide (Al₂O₃)—20 grit

Note X6.2—Perform incoming inspection on every lot of aluminum oxide received by pulling an approximate 50-g sample from every fifth bag or box of the lot. Perform a particle size analysis in accordance with Test Methods D1921 using the following sieve sizes: 12, 16, 18, 20, 25, and pan. The specification for the aluminum oxide shall be as specified in Table X6.1.

X6.6. Test Specimen

X6.6.1 Test specimen sheets prepared from powder or granules shall be 6.4 ± 0.64 mm (0.25 ± 0.025 in.) thick and molded in accordance with Procedure C of Annex A1 of Practice D4703.

X6.6.2 Test specimens (Fig. X6.1) are machined from the sheets. Dimensions of the test specimens shall be 69.85 ± 0.64 mm by 25.4 ± 0.64 mm by 6.35 ± 0.64 mm (2.75 ± 0.025 in. by 1.00 ± 0.025 in. by 0.25 ± 0.025 in.). Each specimen has a large and a small hole; 8.7 mm ($\frac{11}{32}$ in.) diameter and 3.6 mm ($\frac{9}{64}$ in.) diameter drill bits, respectively.

X6.7. Conditioning

X6.7.1 Conditioning—Condition the notched specimens at 23 ± 2 °C for not less than 40 h prior to test.

X6.7.2 *Test Conditions*—Conduct the test in the standard laboratory atmosphere of $23 \pm 2^{\circ}$ C.

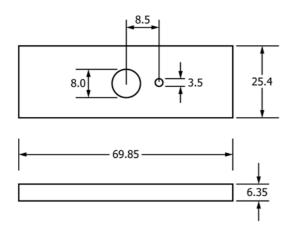
X6.8. Test Procedure

X6.8.1 Weigh each specimen to the nearest 0.0001 g and record this weight at W1. A minimum of two specimens per sample shall be tested.

X6.8.2 Using the pan balance and tared plastic cups, weigh out in separate cups, 450 g of aluminum oxide and 300 g of water. Pour the contents of each cup into the stainless steel testing cup. Repeat procedure for each of the stainless steel testing cups on the unit until all cups contain the aluminum oxide/water mixture.

TABLE X6.1 Specification for Aluminum Oxide

USA Standard	Tyler Equivalent	Sieve Opening	Specification Percent
Sieve Number (mm)	Number	(µm)	Retained
1.70	12	1700	0
1.18	16	1180	20 max
1.00	18	1000	45 min
Contents of 18 + 20	mesh screens-co	ontents of 16 mesh	70 min
	screen		
PAN	PAN	=	3 max



Abrasion Specimen

Nominal	Specimen	dime	nsions
---------	----------	------	--------

	mm	in.
length	69.85	2.75
width	25.4	1.0
thickness	6.35	0.25
large hole diameter	8.0	0.32
small hole diameter	3.5	0.14

FIG. X6.1 Abrasion Test Specimen Dimensions

X6.8.3 Attach the specimens to the stainless steel shafts of the abrasion. Bolt the specimens to the shaft. Do not overtighten the bolts as this will stress the specimen.

X6.8.4 Inspect the gasket in each testing cup to insure it is clean and properly positioned in the groove.

X6.8.5 Push the testing cup upwards, rotating slightly so the specimen is fully immersed in the aluminum oxide/water mixture.

X6.8.6 Move the metal cup support under the cup. Push the support tightly against the cup. Tighten the wing nuts evenly. If not even, it is possible that the cup will rotate or tilt, leading to splattering of the aluminum oxide and result in greater specimen wear

X6.8.7 Set the time to 120 min.

X6.8.8 Raise the hydraulic jack so approximately the bottom one-third of each testing cup is immersed in the water bath. Turn on the water chiller.

X6.8.9 Turn on the main power and push the start button to begin the test.

X6.8.10 When the test unit has shut off at the end of 2 h, turn off the main power and the water chiller. Lower the water bath so the testing cups are no longer immersed. Unscrew the wing nuts from each testing cup and remove the metal cup support. Remove each specimen from the shaft.

X6.8.11 Using your fingernail, carefully remove any melted material that remains attached to the specimen.

X6.8.12 Clean the specimens with water and a brush, and allow them to air-dry for a minimum of 2 h on the vented rack. Insure that no water droplets are inside either of the two holes.

X6.8.13 Reweigh the specimens and record this weight as W2.

X6.9. Calculation

X6.9.1 Calculate the percent of weight loss using the following equation:

% weight loss =
$$[(W1 - W2)/W1] \times 100$$
 (X6.1)

where:

W1 = original weight of test specimen

W2 = weight of test specimen after test

X6.10. Report

X6.10.1 Report the following information:

X6.10.1.1 Complete identification and description of the material tested, including source and manufacturer's code,

X6.10.1.2 Method of sample preparation,

X6.10.1.3 Conditioning of specimens prior to testing,

X6.10.1.4 Length of abrasion time, if other than 120 min,

X6.10.1.5 Specimen number, initial weight of specimen (W1), final weight of specimen after abrasion (W2), and

X6.10.1.6 Percent abrasion for each specimen and average percent abrasion for the sample. Report three significant figures.

X6.11. Precision and Bias

X6.11.1 Table X6.2 is based on a round robin⁴ conducted in 1999 in accordance with Practice E691, involving four materials tested by three laboratories. For each material, all samples were prepared at one source, but the individual specimens were prepared at the laboratories which tested them. Each test result was the result of six individual determinations. Each laboratory obtained 12 test results for each material. (Warning—The explanations of "r" and "R" (X6.11.2 – X6.11.2.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table X6.2 are not to be applied to acceptance or rejection, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots,

TABLE X6.2 Precision Data, Abrasion Index

		Values expressed in Units of %			
Material	Average	S_r^A	$S_R^{B,C}$	r^D	$R^{E,C}$
M-19 IV	2.68	0.12	0.25	0.32	0.69
M-28 IV	2.47	0.16	0.20	0.45	0.55
T-20 IV	2.43	0.16	0.21	0.46	0.59
T-28 IV	2.25	0.10	0.25	0.27	0.71

 $^{{}^{}A}S_{r}$ = Within laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviation of the test results from all of the participating laboratories:

Where

 S_1 = standard deviations of laboratory means.

 $^{\mathcal{O}}$ The calculated \mathcal{S}_{R} and R values in the table are based on data from three laboratories, which is insufficient to provide complete statistical data. However, \mathcal{S}_{R} and R are provided for information only and should not be used in the normal manner.

 D r = within-laboratory critical interval between two test results = $2.8 \times S_{r}$

 $^{^4\,\}mathrm{Supporting}$ data are available from ASTM Headquarters. Request RR:D20-1212.

 $S_r = [[(S_1)^2 + (S_2)^2 \dots + (S_n)^2]/n]^{1/2}$

 $^{^{}B}S_{R}$ = between-laboratories reproducibility, expressed as standard deviation: $S_{R} = [S_{r}^{2} + S_{l}^{2}]^{1/2}$

 $^{^{}E}$ R = between-laboratories critical interval between two test results = 2.8 × S_{R}

formulations, conditions, materials, or laboratories. The principles outlined in Practice E691 are to be used to generate data specific to other materials and laboratories (or between specific laboratories). The principles of X6.11.2 - X6.11.2.3 would then be valid for this data.)

X6.11.2 Concept of "r" and "R" in Table X6.2—If Sr and SR have been calculated from a large enough body of data, and for test results that were averages from testing 6 specimens for each test result, then:

X6.11.2.1 Repeatability—Two results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for the material ("r" is the interval representing the critical difference between two test results for the

same material, obtained by the same operator using the same equipment on the same day in the same laboratory).

X6.11.2.2 *Reproducibility*—Two test results obtained by different laboratories shall be judged not equivalent if they differ by more than the "R" value for that material ("R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories).

X6.11.2.3 Any judgment in accordance with X6.11.2.1 or X6.11.2.2 would have an approximate 95 % (.95) probability of being correct.

X6.11.3 There are no recognized standards by which to estimate bias of this method.



X7. PROPERTIES AND TEST CONDITIONS FOR UHMW-PE

TABLE X7.1 Test Methods Used to Characterize UHMW-PE

Properties	Standard	Specimen Type and Dimensions (mm)	Units	Test Conditions and Supplementary Instructions
Intringia Vigagaity	Specification D4020, Annex A1			Polative vigageity at 0.02 %
Intrinsic Viscosity	Specification D4020, Annex A1	Granules or powder		Relative viscosity at 0.02 % concentration in decalin
	Mechanical Pr			
Tensile Stress at Yield	Test Method D638 and ISO 527-1	Test Method D638, Type 4	MPa	50 mm/min
Tensile Stress at Break		ISO 527-1, Type 2	MPa	
Tensile Elongation at Yield			%	
Tensile Elongation at Break			%	Measured when extensometer is
				used
Nominal Strain at Break Tensile Modulus			% MPa	Gage length is equal to grip distance ASTM 5 mm/min, 2 % secant
				modulus ISO 1 mm/min, chord modulus between 0.05 % and 0.25 s strain
Elongational Stress	Specification D4020, Appendix X5	Specimen specified in Appendix X5		
Flexural Modulus	Test Methods D790	127 by 12.7 by 6.35	MPa	1 % secant, 101.6 mm span, 28 mm min crosshead speed
Double Notched Izod Impact Resistance	Specification D4020, Appendix X2 and Appendix X3	63.5 by 12.7 by 6.35	kJ/m²	
Izod Impact Resistance	Test Method D256	63.5 by 12.7 by 6.35	J/m	
Charpy Impact Resistance	ISO 11542-2, Annex B	120 by 15 by 10	KJ/m ²	
Hardness	Test Method D2240, ISO 868	13 by 13 by 6.35 mm	D	Reading taken after 15 s
Tialuliess	,			neading taken after 13 s
Deflection Temperature under Flexural Load	Thermal Pro Test Methods D648	perties 127 by 12.7 by 6.35	°C	0.454 MPa stress, 0.25 mm
Deficition Temperature under Flexural Load	rest Methods D040	127 by 12.7 by 6.35	C	deflection, 2°C/min heating rate
Thermal Stress Cracking	Test Methods D2951	$125 \times 6.4 \times 1.25$	h	6.4 mm diameter mandrels at 100°C
Melting Temperature	ISO 3146	powder	°C	Method C, ramp rate 10°C/min
Coefficient of Linear Expansion	ISO 10350	Cut from ISO 3167 multipur-	1/°C	TMA, secant value over the tempera
•		pose bar		ture range of +23°C to +55°C
Flammability	Test Method D635, IEC 60695-11-10	125 by 13 by 3	mm/min	Linear burning rate of specimens in horizontal position
	Performance P	roperties		
Abrasion Index	Specification D4020, Appendix X6	69.8 by 25.4 by 6.35	%	1750 RPM, 2 h in aluminum oxide/ water slurry
	Electrical Pro	perties		
Relative permittivity	IEC 250	>60 by >60 by 1		Frequency 100 Hz and 1 MHz (com
Dissipation factor	IEC 250	>60 by >60 by 1		pensate for electrode edge effect) Frequency 100 Hz and 1 MHz (com
Volume resistivity	IEC 93	>60 by > 60 by 1	0	pensate for electrode edge effect)
		>60 by >60 by 1	Ω-m	Voltage 100 V
Surface resistivity	IEC 93	>60 by >60 by 1	Ω	Voltage 100 V
Dielectric (Electrical) Strength	IEC 243-1	>60 by >60 by 1 >60 by >60 by 3	KV/IIIII	Use 25 mm/75 mm coaxial-cylinder electrode configuration. Immerse in IEC 296 transformer oil. Use short time (rapid rise) test
Comparative Tracking Index (CTI)	IEC 112	>15 by >15 by 4		Use solution A
	Miscellaneous F			
Density	Test Methods D792, ISO 1183	50 by 12.7 by 6.35 (ASTM) 10 by 10 by 4 (ISO)	g/cm ³	Density by displacement
Bulk Density	Test Methods D1895	Granules or powder	g/cm ³	
Pourability Angle of Repose	Test Methods D1895 Test Method C1444	Granules or powder Granules or powder	S Angle in	
Coefficient of Friation	Tost Mathod D1904 ISO 9005	63 5 by 63 5 by 6 35	degrees	
Coefficient of Friction Water Absorption	Test Method D1894, ISO 8295 ISO 62	63.5 by 63.5 by 6.35 50 by 50 by 3 or 50 dia. by 3 disc	%	UHMW-PE against metal 24 h immersion
Yellowness Index	Practice E313	50 by 50 by 6.35		2 degree observer, Illuminant C, specular included, UV included, hemispherical configuration

X7.1 Table X7.1 is a listing of additional test methods that can be used to characterize UHMW-PE.



X7.1.1 Test Specimen

X7.1.1.1 Test specimen sheets shall be prepared from powder or granules and molded in accordance with the conditions found in Table X7.2.

TABLE X7.2 Molding Conditions for UHMW-PE Impact Test Specimens

Molding pressure 6.9 to 10.3 MPa Platen temperature 196 to 210 °C Heating time 20 min at 196 to 210 °C Platen cooling rate 15 \pm 2 °C/min from 150 to 90 °C Platen temperature for demolding <30 °C

X7.1.1.2 Specimen Dimensions—Specimen dimensions shall conform to the requirements of the individual tests.

X7.1.1.3 *Conditioning*—Condition the molded specimens at 23 ± 2 °C for not less than 40 h prior to test.

X7.1.1.4 *Test Conditions*—Conduct the tests in the standard laboratory atmosphere of 23 ± 2 °C.

X7.2. Referenced Documents

X7.2.1 ASTM Standards:²

C1444 Test Method for Measuring the Angle of Repose of Free-Flowing Mold Powders (Withdrawn 2005)⁵

D256 Test Methods for Determining the Izod Pendulum Impact Resistance of Plastics

D635 Test Method for Rate of Burning and/or Extent and Time of Burning of Plastics in a Horizontal Position

D638 Test Method for Tensile Properties of Plastics

D648 Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position

D790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D1894 Test Method for Static and Kinetic Coefficients of Friction of Plastic Film and Sheeting

D1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials

D2240 Test Method for Rubber Property—Durometer Hardness

D2951 Test Method for Resistance of Types III and IV Polyethylene Plastics to Thermal Stress-Cracking (Withdrawn 2006)⁵

E313 Practice for Calculating Yellowness and Whiteness Indices from Instrumentally Measured Color Coordinates

X7.2.2 ISO Standards:³

ISO 62 Plastics—Determination of Water Absorption

ISO 527-1 Determination of tensile Properties, Part 1 General Principles

ISO 868 Plastics and Ebonite-Determination of Indention Hardness by Means of a Durometer (Shore Hardness)

ISO 1183 A Plastics—Methods for Determining the Density and Relative Density of Non-cellular Plastics

ISO 1628-3 Plastics—Determination of Viscosity Number and Limiting Viscosity Number—Part 3: Polyethylenes and Polypropylenes

ISO 3146 Plastics—Determination of Melting Behavior (Melting Temperature or Melting Range) of Semicrystalline Polymers

ISO 8295 Plastics—Film and Sheeting-Determination of Coefficients of Friction

ISO 10350 Plastics—Acquisition and presentation of Comparable Single-Point Data

ISO 11542–2 Plastics—Ultra High Molecular-Weight Polyethylene (PE-UHMW) Moulding and Extrusion Materials—Part 2: Preparation of Test Specimens and determination of Properties

X7.2.3 IEC Standards:³

IEC 93 Recommended Methods of Test for Volume and Surface Resistivities of Electrical Insulating Materials

IEC 112 Recommended Method for Determining the Comparative Tracking Index of Solid Insulation Materials

IEC 243-1 Recommended Methods of Test for Electric Strength of Solid Insulating Material at Power Frequencies

IEC 250 Recommended Methods for Determination of the Permittivity and Dielectric Dissipation Factor of Electrical Insulation Materials at Power, Audio, and Radio Frequencies Including Metre Wavelengths

IEC 60695-11-10 Fire Hazard Testing, Part 11–10 Test Flames 50 W Horizontal and Vertical Flames

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4020 - 05) that may impact the use of this standard. (September 1, 2011)

- (1) Updated and added ISO references where appropriate.
- (2) Removed references to Ciba-Geigy as the sole source for stabilizers.
- (3) Updated standard conditioning and test condition requirements.
- (4) Amended permissive language.
- (5) Clarified equations for nominal viscosity average molecular weight.
- (6) Revised procedure for dilute solution viscosity to reflect current practice.

 $^{^{5}\,\}mathrm{The}$ last approved version of this historical standard is referenced on www.astm.org.



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