Designation: D4020 – 11

Standard Specification for Ultra-High-Molecular-Weight Polyethylene Molding and Extrusion Materials[1]

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 classification 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 (ηred 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.

***A Summary of Changes section appears at the end of this standard**

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

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

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

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

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

where:

k = kinetic energy correction constant for the particular viscometer used,
t_s = flow time of solution at 135°C, s,
t_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 ± 0.1°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:

$$\eta_r = (t_s - k/t_s)/(t_o - k/t_o) \quad \text{(A1.1)}$$

where:

k = kinetic energy correction constant for the particular viscometer used,
t_s = flow time of solution at 135°C, and
t_o = 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 ± 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 ± 2° 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, perpendicularity, and offset of the two notches. One of the possible measurement methods is given in Appendix X3.

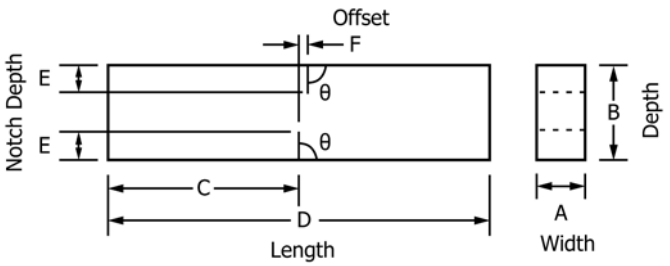

FIG. X2.1 Dimensions of Double-Notched Izod Test Specimens

X2.6. Conditioning

X2.6.1 *Conditioning*—Condition the notched specimens at 23 ± 2°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 ± 2°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

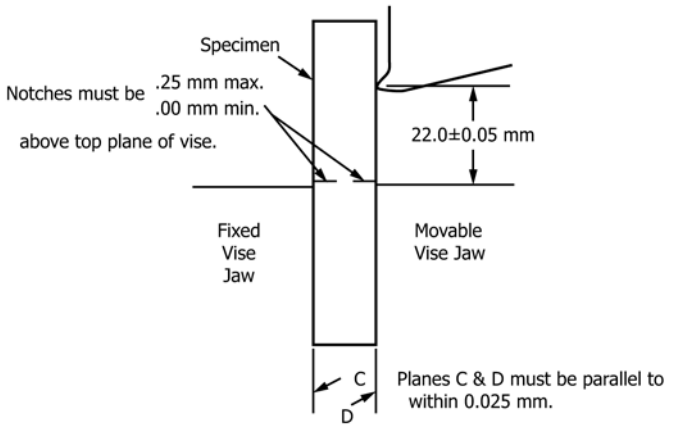

FIG. X2.2 Relationship of Vise, Specimen, and Strike Edge to One Another

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/m^2;

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, dl/g	Values, kJ/m^2				
		Mean	S_r[A]	S_R[B]	I_r[C]	I_R[D]
A	24	128.0	6.5	27.6	18.4	78.2
B	27	120.0	5.4	25.8	15.2	73.1
C	22	103.9	4.1	21.2	11.6	59.9
D	28	56.1	2.2	9.6	6.2	27.2
E	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 60×, 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 the notches to the edges is measured (in this case Line $\overline{AE}$). 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.

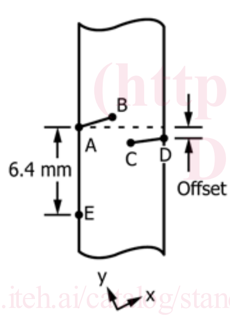

FIG. X3.1 Notch Geometry of Double-Notched Izod Specimen

X3.4 Calculation

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

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

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

$$m = \frac{y_2 - y_1}{x_2 - x_1} \quad (X3.2)$$

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:

$$\text{offset} = |AD| \cos \angle DAE \quad (X3.4)$$

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.