



Designation: D 4020 – 00a

# Standard Specification for Ultra-High-Molecular-Weight Polyethylene Molding and Extrusion Materials<sup>1</sup>

This standard is issued under the fixed designation D 4020; 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 Department of Defense.*

## 1. Scope \*

1.1 This specification provides for the identification of virgin, unmodified 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 It is not intended to differentiate between various molecular weight grades of ultra-high-molecular-weight polyethylene commercially available.

1.3 It is not the function of this specification to provide specific engineering data for design purposes.

1.4 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.

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

1.6 The following precautionary caveat pertains only to the test method portion. Section 7, 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—There is no ISO equivalent specification. However, in ISO 11542-1, a range of viscosity numbers defines the viscosity of UHMW-PE grades. The viscosity numbers are determined in accordance with ISO 1628-3.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 883 Terminology Relating to Plastics<sup>2</sup>

D 1601 Test Method for Dilute Solution Viscosity of Ethylene Polymers<sup>2</sup>

D 1898 Practice for Sampling of Plastics<sup>3</sup>

### 2.2 ISO Standards:<sup>4</sup>

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 D 883.

### 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 ( $\eta_{sp}$ ), reduced viscosity ( $\eta_{red}$  or R.S.V.), intrinsic viscosity ( $\eta$  or I.V.), and the approximate viscosity average molecular weight of virgin resin. The solution viscosity test on thermally processed material is invalid due to inadequate solubility and possible crosslinking.

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

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

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

$$\text{Intrinsic viscosity} = [\eta] = (2\eta_{sp} - 2 \ln \eta_{rel})^{1/2} \div c$$

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 08.01.

<sup>3</sup> Discontinued; see *1997 Annual Book of ASTM Standards*, Vol 08.01.

<sup>4</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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

limiting viscosity number at 0 % concentration  
 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 viscosity average molecular weights. Refer to Appendix X3 for the other equations and their relationship to the 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 viscosity average molecular weight.

#### 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, granules, or pellets.

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 may 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 may 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, the material shall be sampled in accordance with the procedure described in the general and specific sampling procedures of Practice D 1898. Adequate statistical sampling prior to packaging shall be considered an acceptable alternative.

#### 7. Test Method

7.1 *Dilute Solution Viscosity*—Use Test Method D 1601, as modified in Annex A1.

#### 8. Keywords

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

### 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. The relative solution viscosity may be calculated 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*,  $135 \pm 0.1^\circ\text{C}$ , 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*, freshly distilled.
- A1.3.2 *Tetrakis* [methylene 3-(3',5'-di-*tert*-butyl-4'-hydroxyphenyl) propionate] methane.<sup>5</sup>
- A1.3.3 *Xylene*, industrial-grade.
- A1.3.4 *Sulfuric Acid-Potassium Dichromate Cleaning Solution*—To 35 mL of a saturated solution of potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ), carefully add 1 L of concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ).
- A1.3.5 *Acetone*, reagent grade.

##### A1.4 Procedure

- A1.4.1 *Decahydronaphthalene Preparation*—Distill in accordance with Test Method D 1061 and add 0.2 % tetrakis [methylene 3-(3',5'-di-*tert*-butyl-4'-hydroxyphenyl) propionate] methane.
- A1.4.2 *Cleaning the Viscometer*—Clean the viscometer thoroughly with the cleaning solution, wash several times with distilled water, rinse with acetone, and purge with dry nitrogen.

<sup>5</sup> The antioxidant (Irganox® 1010) is available from Ciba-Geigy, Ardsley, NY.

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 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 decahydronaphthalene, and allow the viscometer and solvent to come to thermal equilibrium at 135 ± 0.1°C. Determine the viscosity of the solvent. Remove the decahydronaphthalene with vacuum and wash the viscometer with 200 mL of warm (110 to 120°C) xylene. Remove with vacuum and aspirate dry air or nitrogen to dry the viscometer (2 or 3 min). 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 A1.1) 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<sub>2</sub>SO<sub>4</sub> – K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> cleaning solution.

NOTE A1.1—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:

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

where:

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

*t<sub>s</sub>* = flow time of solution at 135°C, and

*t<sub>o</sub>* = flow time of pure solvent at 135°C.

APPENDICES

(Nonmandatory Information)

X1. IMPACT TEST METHOD FOR ULTRA-HIGH-MOLECULAR-WEIGHT POLYETHYLENE

X1.1 **Scope**

X1.1.1 This test method covers determination of the impact strength of UHMW-PE, which is extremely impact resistant. When tested in accordance with Test Method D 256, 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 D 256 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 D 256 before attempting to use this test method.

X1.1.2 The values stated in SI units are to be regarded as the standard.

X1.1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE X1.1—There is currently no ISO standard that duplicates this test method. The impact strength of UHMW-PE is measured by a double-notched Charpy impact test in the pending ISO/CD 11542-2.

X1.2 **Referenced Documents**

X1.2.1 *ASTM Standards:*

D 256 Test Method for Determining the Pendulum Impact Resistance of Notched Specimens of Plastics<sup>2</sup>

X1.2.2 *ISO Standards:*<sup>4</sup>

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

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

X1.3 **Apparatus**

X1.3.1 The Izod-type impact machine that conforms to the requirements of Test Method D 256, including the calibration and checking methods, shall be used.

X1.4 **Test Specimen**

X1.4.1 The geometry and dimensions of the specimen are given in Fig. X1.1.

X1.4.2 The specimens shall be cut from a sheet compression molded under the following conditions:

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

X1.4.3 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.

X1.4.4 Each specimen shall be free of twist and shall be bounded by mutually perpendicular pairs of plane parallel

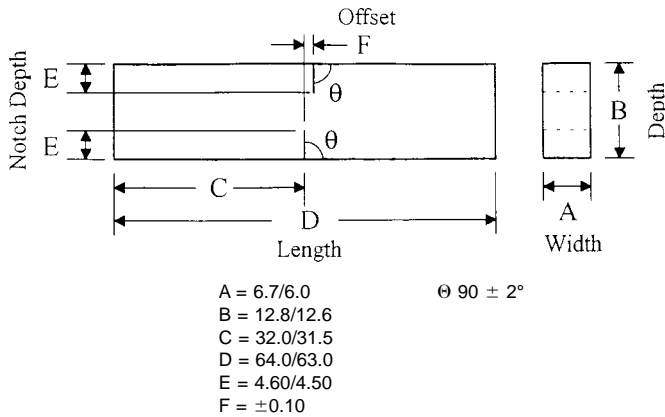


FIG. X1.1 Dimensions of Double-Notched Izod Test Specimens

surfaces, free from scratches, pits, and sink marks.

**X1.5 Notching of Specimens**

X1.5.1 Notching shall be performed on the side parallel to the direction of the application of molding pressure.

X1.5.2 Notching shall be performed in a suitable machine by pressing in a  $0.23 \pm 0.03$ -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.

X1.5.3 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 X2.

**X1.6 Conditioning**

X1.6.1 *Conditioning*—Condition the notched specimens at  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  relative humidity for not less than 40 h prior to test.

X1.6.2 *Test Conditions*—Conduct the test in the standard laboratory atmosphere of  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  relative humidity.

**X1.7 Procedure**

X1.7.1 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 X1.6.

X1.7.2 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.

X1.7.3 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.

X1.7.4 Before testing the specimens, perform the following operations on the machine:

X1.7.4.1 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*.

X1.7.4.2 Without resetting the pointer, raise the pendulum and release again. The pointer should move up the scale an additional amount. Repeat this procedure until a swing causes no additional movement of the pointer, and note the final reading as one reading of Factor *B*.

X1.7.4.3 Repeat the above two operations several times, and calculate and record the average *A* and *B* readings.

X1.7.5 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. X1.2. Note that the top plane of the vise shall be  $0.13 \pm 0.13$  mm below the notches.

X1.7.6 Release the pendulum and note and record the excess energy remaining in the pendulum after breaking the specimen.

X1.7.7 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 D 256. Subtract the correction so calculated from the 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 X1.7.2 to obtain its impact strength in kilojoules per square metre.

X1.7.8 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.

X1.7.9 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

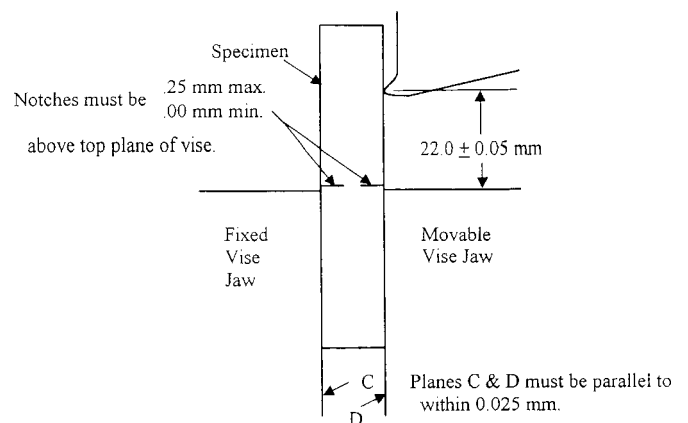


FIG. X1.2 Relationship of Vise, Specimen, and Strike Edge to One Another

completely. The results obtained from unbroken specimens shall be considered a departure from standard and shall not be reported as a standard result.

**X1.8 Report**

X1.8.1 Report the following information:

X1.8.2 Complete identification of the material tested, including type, source, manufacturer’s lot number, and previous history;

X1.8.3 Compression molding conditions;

X1.8.4 Capacity of the pendulum, J;

X1.8.5 Total number of specimens tested;

X1.8.6 Number of those specimens that result in complete break;

X1.8.7 Average impact strength, kJ/m<sup>2</sup>;

X1.8.8 Standard deviation; and

X1.8.9 Percent of specimens failing in each category, suffixed by the corresponding letter code from X1.7.8.

**X1.9 Precision and Bias**

X1.9.1 Table X1.1 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.

**TABLE X1.1 Precision of the Double-Notched Izod Impact Test Method**

Material	Intrinsic Viscosity, dl/g	Values, kJ/m <sup>2</sup>				
		Mean	S <sub>r</sub> <sup>A</sup>	S <sub>R</sub> <sup>B</sup>	I <sub>r</sub> <sup>C</sup>	I <sub>R</sub> <sup>D</sup>
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

<sup>A</sup>S<sub>r</sub> = within-laboratory standard deviation of the average.

<sup>B</sup>S<sub>R</sub> = between-laboratories standard deviation of the average.

<sup>C</sup>I<sub>r</sub> = 2.83 S<sub>r</sub>.

<sup>D</sup>I<sub>R</sub> = 2.83 S<sub>R</sub>.

X1.9.1.1 *Repeatability*, I<sub>r</sub> (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 should be judged not equivalent if they differ by more than the I<sub>r</sub> value for that material.

X1.9.1.2 *Reproducibility*, I<sub>R</sub> (Comparing two test results for the same material, obtained by different operators using different equipment on different days)—The two test results should be judged not equivalent if they differ by more than the I<sub>R</sub> value for that material.

**X2. MEASUREMENT METHOD OF IMPERFECTIONS IN SPECIMEN NOTCHING**

X2.1 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. X2.1).

**X2.2 Apparatus**

X2.2.1 *Reflective Optical Microscope*, ocular, 40 to 60×, with an X-Y stage accurate to 0.0025 mm.

X2.2.2 *Eyepiece*, with a crosshair.

X2.2.3 *Fiber Optic Illumination*.

**X2.3 Procedure**

X2.3.1 Lay the specimen on one of its sides and mount it securely on the X-Y stage.

X2.3.2 The beginning and ending points of the notches are labeled from A to D in Fig. X2.1. 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.

X2.3.3 Both the microscope and the base of the X-Y stage should be 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.

**X2.4 Calculation**

X2.4.1 The following equation is used to calculate the perpendicularity of the notches:

$$\angle EAB = \tan^{-1} \frac{m_2 - m_1}{1 + m_2 m_1} \tag{X2.1}$$

where:

m<sub>1</sub> and m<sub>2</sub> = slopes of line  $\overline{AE}$  and  $\overline{AB}$  with respect to the coordinate system.

m<sub>1</sub> and m<sub>2</sub> are calculated from

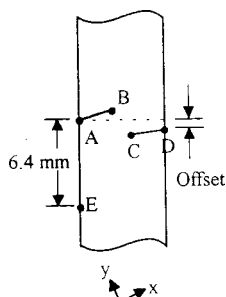
$$m = \frac{y_2 - y_1}{x_2 - x_1} \tag{X2.2}$$

where:

m = slope, and  
(x<sub>1</sub>, y<sub>1</sub>) and (x<sub>2</sub>, y<sub>2</sub>) = coordinates of the end points of the line.

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

$$I = \sqrt{(x_2 - x_1)^2 + (y_2 - y_1)^2} \tag{X2.3}$$



**FIG. X2.1 Notch Geometry of Double-Notched Izod Specimen**

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

$$\text{offset} = |\overline{AD}| \cos \angle DAE \quad (X2.4)$$

### X3. VISCOSITY AVERAGE MOLECULAR WEIGHT OF ULTRA-HIGH MOLECULAR-WEIGHT POLYETHYLENE

#### X3.1 Scope

X3.1.1 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.

X3.1.2 The dilute viscosity test method may provide satisfactory correlations for viscosity average molecular weight within a specific manufacturing process, but may not apply for

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

X3.1.3 Fig. X3.1 shows the relationship between the five equations with respect to viscosity average molecular weight versus intrinsic viscosity.

X3.1.4 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.

#### SUMMARY OF CHANGES

This section identifies the location of selected changes to this specification. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this specification. This section may include descriptions of the changes or reasons for the changes, or both.

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- (1) Added Note 1, ISO equivalency statement.
- (2) In 1.4, changed relative viscosity from 2.30 to 1.44.
- (3) In 3.2.1, changed relative viscosity from 2.30 to 0.05 % concentration to 1.44 at a concentration of 0.02 % for polymer in solvent.

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- (1) Changed “can” to “shall” in 3.2.1.1
- (2) Changed “weight” to “viscosity” in 3.2.1.1
- (3) Changed “nominal molecular weight” to “nominal viscosity molecular weight” in the equations in 3.2.1.1.
- (4) Add Note 2 in 3.2.1.1.

- (5) Changed “0.05 g” to “0.02 g” in A1.1.1.
- (6) Changed “35 to 43” to “14 to 17”, “1.8” to “4.5”, “36” to “15,” and “64.8” to “67.5” in A1.4.3. Changes made to achieve correct concentration.
- (7) Added Appendix X3 covering UHMWPE molecular weight.

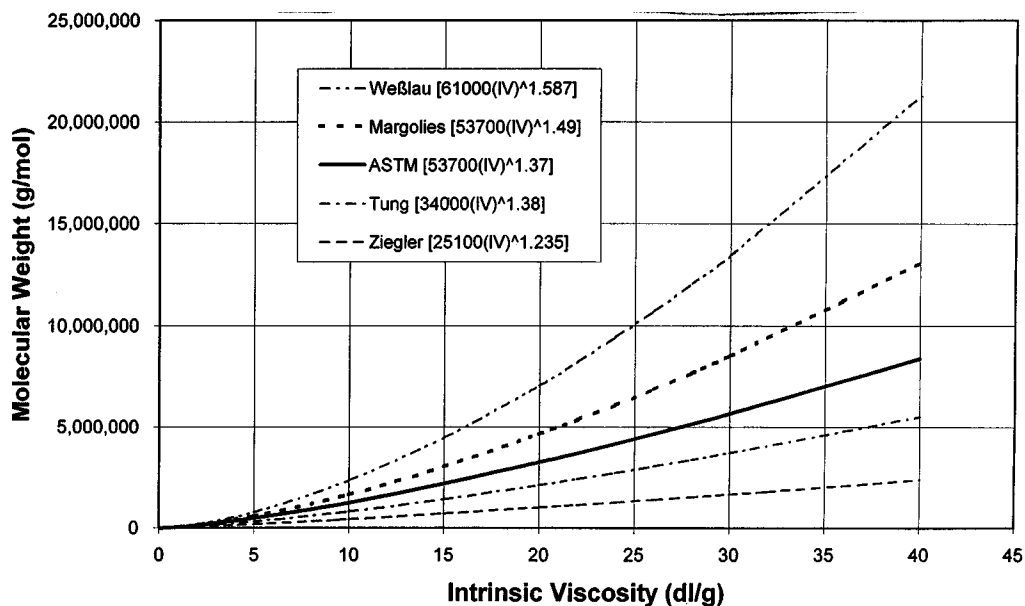


FIG. X3.1 Known Molecular Weight Equations (Correlating with Intrinsic Viscosity)

 **D 4020**

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