Designation: D4020 – 18

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 (UHMWPE) 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, health, and environmental 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.

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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 UHMWPE resins. The following calculations shall be used to approximate the specific viscosity

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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**

(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:

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

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

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

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 X2 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 UHMWPE 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.

5.4 Additional test methods and conditions that are commonly used to characterize UHMWPE are listed in Table X4.1.

5.4.1 Refer to Annex A2 for requirements regarding specimen preparation, dimensions, and conditioning requirements for these tests.

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; UHMWPE; viscosity

ANNEXES

(Mandatory Information)

A1. DILUTE SOLUTION VISCOSITY

A1.1 General Description

A1.1.1 The test sequence consists of dissolving UHMWPE 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 UHMWPE in a vacuum oven for 2 h at 60°C. Weigh 14 to 17 mg of the dry UHMWPE onto a slide cover slip. Use the buret to transfer the stabilized decahydronaphthalene at room temperature into the Erlenmeyer flask, measuring, in milliliters, a volume equal to 4.5 times the UHMWPE weight in milligrams, for example, 15 mg of UHMWPE and 67.5 mL of decahydronaphthalene. Heat the decahydronaphthalene, with stirring, to 150°C, and drop in the UHMWPE 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 UHMWPE 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.

A2. TEST SPECIMEN PREPARATION, DIMENSIONS, AND CONDITIONING REQUIREMENTS

A2.1 *Test Specimens*

A2.1.1 Test specimen sheets shall be prepared from powder or granules and molded in accordance with 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
Below 90°C	Maintain pressure and cool as quickly as possible to <30°C
Platen temperature for demolding	<30°C

A2.2 *Specimen Dimensions*

A2.2.1 Specimen dimensions shall conform to the requirements of the individual tests.

A2.3 *Conditioning*

A2.3.1 Condition the notched specimens at 23 ± 2°C for not less than 16 h prior to test.

A2.4 *Test Conditions*

A2.4.1 Conduct the test in the standard laboratory atmosphere of 23 ± 2°C.