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Standard Test Method for Melting Point of Semicrystalline Polymers by the Hot Stage Microscopy Method¹

This standard is issued under the fixed designation D 2117; 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 test method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

^{ε1} NOTE—The safety hazards caveat was added editorially to the Scope and Sections 3 through 5 were renumbered in March 1988.

1. Scope

1.1 This test method covers the determination of the melting point of semicrystalline polymers (Note 1).

NOTE 1—Another procedure for the melting point of nylon resins may be found in Specification D 789.

1.2 This test method is applicable to semicrystalline polymers in granular or sheet form, 0.04 mm (1.6 mils) or less in thickness. The normal operating temperature range is 30 to 230°C. A maximum operating temperature of 350°C can be obtained with special equipment (see 7.2).

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

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 618 Methods of Conditioning Plastics and Electrical Insulating Materials for Testing²

D 789 Specification for Nylon Injection Molding and Extrusion Materials²

D 1898 Practice for Sampling of Plastics³

E 77 Method for Verification and Calibration of Liquid-in-Glass Thermometers⁴

3. Terminology

3.1 *semicrystalline polymers*—those polymers capable of forming at least two-dimensional intermolecular order as prepared or as developed by suitable thermal treatments. The amount and degree of perfection of the ordering or crystallization depends on the spatial relationship of the atoms in the

molecules, as well as the thermal or other treatment.

4. Summary of Test Method

4.1 This test method consists of heating the sample by a hot stage unit mounted under a microscope and viewing it between crossed polars. When the crystalline material melts, the characteristic double refraction from the crystalline aggregates disappears. The point at which the double refraction or birefringence completely disappears is taken as the melting point of the polymer. The effect of exposure to heat or air, or both, on the melting point can be detected by the use of this test method.

5. Significance and Use

5.1 This test method is useful for determining the melting point of semicrystalline polymers where a high degree of precision is desired (see Section 14).

5.2 The test method is suitable for specification acceptance, manufacturing control, development, or research.

6. Interferences

6.1 An appreciable increase in particle size above that specified will result in a large melting range and obscure the sharp end point. The presence of anisotropic crystals, for example, some inorganic substances, will reduce the accuracy of the melting point due to the persistence of their double refraction after the greater part of the sample has melted.

6.2 In some cases heat and air or heat alone will cause changes in the melting point. Where it has been shown that there is an effect of heat and air on the melting point, provision is made in 12.3 for an inert gas blanket. In the case of heat alone below the melting point (solid state polymerization) or at the melting point (further polymerization or degradation), the effect of heat should be determined by remelting a sample that has already been run. If the two determinations check within $\pm 1^\circ\text{C}$, the procedure as outlined may be followed. If the material is affected by heat alone, below or at the melting point, the melting point of the material cannot be determined by this test method.

7. Apparatus

7.1 *Compound Microscope*, with magnifications from 50 $\times$ to 100 $\times$ and at least a 6-mm working distance. A cap analyzer and disk polarizer to fit the microscope are re-

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties (Section D20.30.07).

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 08.02.

⁴ *Annual Book of ASTM Standards*, Vol 14.01.