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An American National Standard

Standard Test Method to Determine Melting Temperature of Synthetic Fibers[1]

This standard is issued under the fixed designation D7138; 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.

1. Scope

1.1 Either of two test methods are used to determine the melting temperature of thermoplastic fibers, yarns, or threads.

1.2 Method 1 can be used to determine melting temperatures for blends of multiple fiber material types. Method 2 can only be used to determine the melting temperature of a single fiber material type.

1.2.1 Method 1, Differential Scanning Calorimetry (DSC), measures changes in heat capacity and will detect the glass transition, the crystalline melting and endothermic thermal degradation.

1.2.2 Method 2, a visual determination of melting, determines any change that visually appears as a transition from a solid to a liquid state.

1.2.3 Due to the differences in what each test method measures, the results from Method 1 and Method 2 cannot be compared.

1.3 The values stated in either SI units or other units are to be regarded separately. The values stated in each system are not exact equivalents; therefore, each system shall be used independently without combining values.

1.4 *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.*

2. Referenced Documents

~~2.1 *ASTM Standard:*[2]~~
~~D123 Terminology Relating to Textiles~~
~~D1776 Practice for Conditioning and Testing Textiles~~
~~D2257 Test Method for Extractable Matter in Textiles~~
~~D3333 Practice for Sampling Manufactured Staple Fibers, Sliver, or Tow for Testing~~

2.1 *ASTM Standards:*[2]
D123 Terminology Relating to Textiles
D276 Test Methods for Identification of Fibers in Textiles
D1776 Practice for Conditioning and Testing Textiles
D2257 Test Method for Extractable Matter in Textiles
D2258 Practice for Sampling Yarn for Testing
D3333 Practice for Sampling Manufactured Staple Fibers, Sliver, or Tow for Testing

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *melting temperature, n*—the temperature or range of temperatures at which a substance is observed to transition to a liquid-like state.

3.1.2 For all other terminology related to textiles, see Terminology D123.

4. Summary of Test Method

4.1 *Method 1:*

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[1] This test method is under the jurisdiction of ASTM Committee D13 on Textiles and is the direct responsibility of Subcommittee D13.51 on ~~Conditioning and,~~ Conditioning, Chemical and Thermal Properties.

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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.

4.1.1 A specimen of fiber and a reference sample are positioned into the designated heating blocks of a Differential Scanning Calorimetry (DSC) instrument.

4.1.2 Use the DSC data to determine the fiber specimen melting temperature.

4.2 *Method 2:*

4.2.1 A specimen of fiber is positioned in a melting temperature device.

4.2.2 The temperature of the device is raised until the fiber specimen reaches its melting temperature as determined by visual observation.

5. Significance and Use

5.1 Either of these two test methods is used to determine the temperature at which a synthetic fiber specimen changes from a solid to a liquid-like state.

5.1.1 Synthetic fibers may be either amorphous or semi-crystalline thermoplastics or thermosets. Synthetic fibers may change from the solid to a liquid-like state on heating because of the glass transition of amorphous polymers, the melting of crystalline regions of semi-crystalline polymers, or at the onset of degradation. Thermoplastic fibers consist of crystalline and amorphous regions and may be manufactured with a range of molecular weights. The amorphous and crystalline fiber structure and variable molecular weight can lead to a melting temperature range instead of a discreet melting point (see Table X1.1).

5.2 This test method is considered satisfactory for acceptance testing of commercial shipments.

5.2.1 If there are differences of practical significance between reported test results for two or more laboratories, perform comparative testing to determine if there is a statistical bias between them, using competent statistical assistance. As a minimum, use the samples for such a comparative test that are as homogeneous as possible, drawn from the same lot of material as the samples that resulted in disparate results during initial testing and randomly assigned in equal numbers to each laboratory. Compare the test results from the laboratories involved using a statistical test for unpaired data, at a probability level chosen prior to the testing series. If bias is found, either its cause must be found and corrected, or future test results for that material must be adjusted in consideration of the known bias.

5.3 This test method is suitable for quality control testing of synthetic fibers and product comparisons of different fibers by manufacturers, retailers, and users.

5.4 If the test method is used to identify fiber material type, it is important to test a known reference material at the same laboratory with the same test method to confirm the fiber identification. In addition, since some fiber types have similar melting temperatures or overlapping melting temperature ranges as show in Table X1.1, secondary methods for fiber identification as described in Test Methods D276 will be required to make fiber identifications.

6. Apparatus

6.1 *Apparatus for Method 1, using a DSC instrument:*

6.2 *Apparatus for Method 2:*

6.2.1 *Test Unit Configuration*—Apparatus used consists of an electrically heated stage which has a temperature range from 20ºC (68ºF) to 300ºC (572ºF) or at least 10ºC (18ºF) above the highest melting temperature to be measured. See Fig. X1.1 for a typical apparatus.

6.2.1.1 The adjustment mechanism shall be able to control the heat input into the stage.

6.2.1.2 Thermometer for measuring temperature shall be accurate to +0.5ºC (+1ºF).

6.2.1.3 The device shall have a low powered magnifying glass to permit visual examination.

6.2.1.4 The device shall have a top and bottom microcover glass. The top glass shall fit directly over the bottom glass so that the fiber specimen rests between the microcovers.

6.2.2 Soxhlet extraction device. See Fig. X1.2.

7. Preparation of Apparatus

7.1 *Lot Sample:* As a lot sample for acceptance testing, take at random the number of laboratory sampling units as directed in an applicable material specification or other agreement between the purchaser and the supplier. In the absence of a material specification or other agreement, use Practice D2258 or Practice D3333, as applicable. Consider containers, such as cartons, bales or other shipping containers to be the primary sampling units.

7.2 *Laboratory Sample Unit:* As a laboratory sampling unit for acceptance testing, take as directed in Practice D2258 or Practice D3333, as applicable. If the fiber sample requires cleaning using a Soxhlet extractor proceed to 7.2.1. If cleaning using a Soxhlet extractor is not required proceed to 7.3.

7.2.1 Place sample in a Soxhlet extractor (see Fig. X1.2) and perform twenty extractions using a chloroform, USP reagent. Follow extraction procedure in D2257. Other solvents such as methanol, ethanol or isopropyl alcohol may be used if they are found to be effective in removing fiber finishes and coatings.

7.2.2 Dry samples and return to equilibrium in accordance with D1776.

7.3 *Test Specimens:*

7.3.1 *Method 1:*

7.3.1.1 *DSC Sample Preparation*—Cut the samples into very small pieces with scissors and put a 5 to 7 mg specimen into the aluminum pan. Make a small hole on the lid with a needle and close the pan with the help of a sample crimper. Make another sample, which will be used as the reference sample by closing an empty pan with lid.

7.3.1.2 Prepare five specimens and five references to perform five replicates.

7.3.2 *Method 2:*

7.3.2.1 From each laboratory sampling unit, cut five fiber samples into specimens having a length of approximately 2 mm (.0625 in).

7.3.2.2 Prepare the five specimens to perform five replicates.

8. Calibration

8.1 Follow the manufacturer's instructions to calibrate the melting temperature apparatus (see Appendix X1).

8.2 Verify the melting temperature apparatus performance by using fiber of a known melting temperature (see Table X1.1).

NOTE 1— Use a fiber having a melting temperature similar to the specimen to be tested (see Table X1.1).
NOTE 2—Avoid using a fiber with a wide melting temperature range as the verification fiber (see Table X1.1).

8.2.1 Verification is achieved when the melting temperature of the verification fiber used is within +1ºC (+2ºF) of its known value.

9. Test Procedure

9.1 *Procedure for Method 1:*

9.1.1 Put the specimen and the reference in the designated heating blocks (in accordance with the instrument manufacturer's manual) inside the heater chamber. Close the chamber, and start heating at the rate of 10°C/min to approximately 50°C above the melting point of the fiber being tested in accordance with Table X1.1.

9.1.2 Record the temperature. Cool the chamber, and repeat the measurement on a new specimen until five specimens are completed.

9.2 *Procedure for Method 2:*

9.2.1 Place a specimen consisting of a sufficient quantity of fibers to observe specimen melting, approximately 50 cut fibers, on the bottom microcover glass of the test apparatus and cover with the top micro glass.

9.2.2 If the fiber melting temperature is known, set the dial of the test apparatus to a temperature 15ºC (+27ºF) below the anticipated melting temperature. Proceed to 9.2.5.

9.2.3 If the melting temperature is not known before testing, it can be determined by using the following approach:

9.2.3.1 Place the specimen in the apparatus and elevate the temperature to 140ºC (284ºF). If the specimen melts, test the next specimen, after cool down of the apparatus, at a temperature 20ºC (36ºF) lower. If the specimen does not melt, proceed to 9.2.5.

9.2.3.2 Continue this process until the fiber does not melt.

9.2.4 Gradually raise the temperature at 3ºC (+5ºF) per minute until the fiber demonstrates melting.

9.2.5 Observe the specimen using the magnifying glass.

9.2.6 Read the temperature to nearest degree C (F) on the test apparatus when it is observed that the solid demonstrates a liquid flow.

10. Final Test Report

10.1 Report that the melting temperature of fibers was determined as directed in Test Method D7138. Indicate whether Method 1 or Method 2 was used to determine the melting temperature. Describe the material or product sampled and the method of sampling used.

10.2 Report the following information for the laboratory sampling unit and for the lot as applicable to a material specification or contract order. For Method 1, if a fiber blend sample is tested, report the results for each fiber type in the blend sample.

10.2.1 Report the melting temperature of each of the five replicates to the nearest degree C (F).

10.2.2 Calculate and report the average melting temperature of the five replicates to the nearest degree C (F).

11. Precision and Bias

11.1 *Precision and Bias for Method 1:*

11.2 *Precision and Bias for Method 2:*

11.2.1 *Precision*—The repeatability standard deviation has been determined to be 1.3ºC for a polyester sample and 2.1ºC for a nylon sample. The 95 % confidence limit for the polyester sample is 240 ± 2ºC and for the 6–6 nylon sample is 238 ± 3ºC. The reproducibility of this test method is being determined and will be available on or before January 31, 2009.