



Standard Test Method for Rubber Chemicals—Melting Range¹

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1. Scope

1.1 This test method covers the determination of the melting range of commercial rubber processing chemicals either by use of capillary melting point tubes or by differential scanning calorimetry (DSC).

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

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.*

2. Referenced Documents

2.1 ASTM Standards:

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²

E 1 Specification for ASTM Thermometers³

E 324 Test Method for Relative Initial and Final Melting Points and the Melting Range of Organic Chemicals⁴

E 473 Terminology Relating to Thermal Analysis⁵

E 967 Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers⁵

3. Terminology

3.1 Definition:

3.1.1 *differential scanning calorimetry*—see Terminology E 473.

4. Significance and Use

4.1 This test method may be used for research and development. It also may be used for quality assurance, provided a standard has been agreed upon between a producer and a user.

4.2 For identification purposes, melting range should be supplemented by measurements of a more specific physical and chemical property.

4.3 This test method is not recommended for rubber chemicals that decompose at their melting ranges.

4.4 The melting range as determined by Method A—Capillary Tube Melting Range is not recommended as a criterion of purity of a rubber chemical.

5. Sampling

5.1 Grind a representative sample of the chemical to be tested with a mortar and pestle, if necessary, to pass completely through a 150 μm (No. 100) sieve. Use the sample without further treatment.

METHOD A—CAPILLARY TUBE MELTING RANGE

6. Apparatus

6.1 *Melting Apparatus*—Any electric melting apparatus that satisfies the requirements of Test Method E 324 may be used or any suitable manually heated oil bath such as Hersberg tube.

6.2 *Capillary Tube*—The capillary tube to contain the sample shall be a glass tube approximately 150 mm long and 1.2 to 1.4 mm in internal diameter with walls 0.2 to 0.3 mm thick and closed at one end.

6.3 *Thermometer*—The thermometer shall be of the partial immersion type and of suitable range selected from Specification E 1, or of an equivalent range as specified by the Chemical Manufacturers Association. It shall be divided into subdivisions of 0.5°C (1°F) or less. Corrections for the thermometer shall be determined by calibration against a thermometer certified by National Institute of Standards and Technology.

6.4 *Sieve*—A 150-μm (No. 100) sieve for screening the sample shall be provided.

7. Procedure

7.1 Select the thermometer of the proper range and support it so that it is immersed to the immersion mark in the liquid of the bath.

7.2 Charge the capillary glass tube with sufficient powder to form a column in the bottom of the tube about 3 to 6-mm high when packed down as closely as possible by moderate tapping on a solid surface.

7.3 Heat the bath until a temperature approximately 25°C below the expected melting range is reached. Then regulate the rate of rise so that it averages about 3°C/min for the rest of the determination except that the rate of rise is $1 \pm 0.2^\circ\text{C}/\text{min}$

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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² Annual Book of ASTM Standards, Vol 09.01.

³ Annual Book of ASTM Standards, Vol 14.03.

⁴ Annual Book of ASTM Standards, Vol 15.05.

⁵ Annual Book of ASTM Standards, Vol 14.02.