

Designation: D7735 - 17 (Reapproved 2022)

Standard Test Method for Type A Durometer Hardness Testing of Thermoplastic Pavement Marking at Elevated Temperatures¹

This standard is issued under the fixed designation D7735; 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 This test method covers Type A Durometer hardness of thermoplastic pavement marking material at elevated temperatures.
- 1.2 This test method is similar to Test Method D2240 for hardness with special attention given to keeping the temperature of the test sample and instrument constant. Within the highway materials community, it is sometimes known as "indentation resistance."
- 1.3 All materials, instruments or equipment used for the determination of temperature, hardness and time shall be traceable to National Institute of Standards and Technology or other internationally recognized organizations parallel in nature.
- 1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.
- 1.5 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.
- 1.6 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

- D16 Terminology for Paint, Related Coatings, Materials, and Applications
- D2240 Test Method for Rubber Property—Durometer Hardness
- D7307 Practice for Sampling of Thermoplastic Pavement Marking Materials
- D7308 Practice for Sample Preparation of Thermoplastic Pavement Marking Materials
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

- 3.1 Definitions:
- 3.1.1 The terms and definitions in Terminology D16 apply to this method.
- 3.1.2 hardness, adv—the value of resistance of a small penetrating device. In the case of the Type A Durometer in accordance with Test Method D2240, the presser foot/indentor (spherical to 0.79 mm \pm 0.03 mm or 0.03 in. \pm 0.001 in.) is connected to a spring loaded to a total of 0.075 N/Durometer units at full deflection. For reading of Type A hardness the instruments scale reads from 0 to 100. Minimal deflection is 0 (softest) and maximum is 100 (hardest).
 - 3.1.3 *thermoplastic*, n—pavement marking (same as 3.1.4).
- 3.1.4 thermoplastic pavement marking—a highly filled 100 % total solids highway marking system that when heated to a molten state can be extruded or sprayed onto a road surface and when cooled forms a solid durable delineator or thermoplastic pavement marking usually melted to 220 °C (425 °F).

4. Summary of Test Method

4.1 This method was developed to enable the testing of hardness or indentation resistance of thermoplastic pavement marking material at ambient roadway/highway temperatures. Due to the speed at which basic thermoplastic pavement marking cools, testing its hardness at elevated temperatures is difficult, even at a relatively low 46 °C (115 °F). Existing non-standard methods are not consistent because they allow the samples to change temperature during testing. By keeping both

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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.

the test samples and the hardness tester in a regulated environment before and during testing, the results are much more repeatable within a laboratory. The goal for this method is to allow results from this test to be more reproducible from laboratory to laboratory.

4.2 The methodology involves measuring the hardness of thermoplastic pavement marking by keeping the test sample in the elevated temperature environment (a water bath) during the testing process and using a Durometer that can be placed in the water bath and function. This method could be performed in a glove box/oven if the door was not opened before or during the testing.

5. Significance and Use

5.1 This method is based on the penetration of a specific type of indentor when forced into the material under specified conditions. The indentation hardness is inversely related to the penetration and is dependent on the elastic modulus, viscoelastic behavior of the material, geometry of the indentor, the applied force and temperature at test. This test method is empirical and therefore primarily for control purposes. No simple relationship exists between indentation hardness determined by this test method and any fundamental property of the material tested.

6. Apparatus

- 6.1 Safety Glasses.
- 6.2 Aluminum Pans (20 mL) or adequate size for tester base.
- 6.3 A Hardness Tester Type A Needle-type Durometer in accordance with Test Method D2240.
 - 6.4 Constant 1000 g Load Weight for Type A Durometer.
- 6.5 Optional—Test Block Kit containing Type A Durometer samples with values traceable to an accredited calibration laboratory.
- 6.6 *Oven* controllable to 46 °C \pm 1.0 °C (115 °F \pm 3.0 °F) (or within 2.5 % of set point).
- 6.7 Water Bath controllable to 46 °C \pm 1.0 °C (115 °F \pm 3.0 °F) (or within 2.5 % of set point). A minimum of filtered water should be used so that minerals in normal tap water cannot settle out and clog the inner workings of the device.
 - 6.8 Digital Timer in seconds.

7. Hazards

7.1 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.

8. Calibration and Standardization

8.1 Calibration of the Type A hardness tester shall be done regularly by following the manufacturers recommendations and following the Calibration section of Test Method D2240. The test block kit can be used to verify that the instrument is working properly.

9. Procedure

- 9.1 Sample the thermoplastic pavement marking in accordance with Practice D7307 then begin preparation of samples in accordance with Practice D7308.
- 9.2 Pour thermoplastic that has been heated to 425 °F (or per manufacturer's recommendation) into a 20 mL aluminum pan, or larger, to approximately half full, and then allow cooling to room temperature.
- 9.3 Turn on heater on water bath and oscillator or stirrer and allow the temperature to stabilize to 46 °C \pm 1.0 °C (115 °F \pm 3.0 °F) or as agreed upon by the involved parties
- 9.4 Turn on oven set to 46 °C \pm 1.0 °C (115 °F \pm 3.0 °F) or as mutually agreed upon by the involved parties and place the Type A Durometer inside

Note 1—A bath or oven operated outside the recommended temperature control may result in greater result variability.

- 9.4.1 Other temperatures can be used when agreed upon between the parties involved.
- 9.5 Place cooled thermoplastic pavement marking hardness sample in water bath.
- 9.6 Samples should be allowed to equilibrate to the required temperature for at least one hour before being tested.
- 9.6.1 Samples should be tested within three hours of being placed into the water.
- 9.7 Before testing sample, remove lid from water bath. Quickly place hardness tester from oven in the water bath making sure that the needle point and bottom of weight is below the waterline. This will ensure that tester is exactly the same temperature as sample.

Note 2—If the tester is removed from the water during the testing process, its temperature will change and cause variations in results. Sample(s) can be tested multiple times as long as sample and tester bottom are not removed from water during testing.

- 9.8 To test the sample, first set the timer so that at least a fifteen second countdown can be counted once the measurement begins.
- 9.9 Place the hardness tester over the sample keeping both tester and sample under water at all times.
- 9.10 Lower the hardness tester over the center of the test sample.
- 9.11 As soon as contact is made, start or check timer and release tester. The tester dial normally moves constantly lower.
- 9.12 When fifteen seconds is reached, note the Type A hardness value on the dial and record.
 - 9.12.1 Make at least two readings then average.

Note 3—Caution should be taken when making multiple readings not to test in the same spot or after the tester has been removed from the water.

Note 4—Care should be taken to ensure that the force is applied vertically to the indentor tip, as lateral force or uneven surface will cause errors in results.

Note 5—Excess water left in the device after testing can cause malfunctioning of the device. Therefore a pressing of the needle on a paper towel after use can allow for the draining of retained water.

TABLE 1 Hardness — Day 1 (Type A units)

Material	Average ^A x	Repeatability Standard Deviation S.	Reproducibility Standard Deviation S _B	Repeatability Limit	Reproducibility Limit
White Alkyd Extrude Thermoplastic Pavement Marking	46.06	2.29	2.36	6.42	6.60
Yellow Alkyd Extrude Thermoplastic Pavement Marking	54.83	2.04	2.08	5.70	5.82

^A The average of the technicians' calculated averages.

TABLE 2 Hardness — Day 2 (Type A units)

Material	Average ^A x̄	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit R
White Alkyd Extrude Thermoplastic Pavement Marking	42.00	1.81	S _R 2.61	5.06	7.30
Yellow Alkyd Extrude Thermoplastic Pavement Marking	49.89	2.32	2.32	6.51	6.51

^A The average of the technicians' calculated averages.

9.13 If sample fails, another reading may be taken immediately, but do not place needle point in the same indentation.

10. Report

- 10.1 Hardness measurements and average (Type A).
- 10.1.1 Date of testing.
- 10.1.2 Oven and water bath temperature.
- 10.1.3 Manufacturer, type and serial number of the Durometer.
 - 10.1.4 Date of last calibration and calibration due date.

11. Precision and Bias³

- 11.1 The precision of this test method is based on an Interlaboratory study of ASTM D7735 Standard Test Method for Hardness of Thermoplastic Pavement Marking, conducted in 2010. A single laboratory, with three technicians, participated in this study. Each of the technicians tested two different materials a total of twelve times over a period of two days. Every "test result" reported represents an individual determination. Except for the use of only a single laboratory, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. RR:D01-1160.
- 11.1.1 Repeatability Limit (r)—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.
 - 11.1.2 Repeatability limits are listed in Table 1 and Table 2.

- 11.1.3 Repeatability Limit (R)—Two test results shall be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.
- 11.1.4 In this study, since three different technicians in a single laboratory performed the analyses, only modified reproducibility limits can be listed in Table 1 and Table 2.
- 11.1.5 The reproducibility limit mentioned above is not used as specified in Practice E177, as it represents the anticipated variability between technicians in the same laboratory.
- 11.1.5.1 Any judgment in accordance with statements 11.1.1 and 11.1.3 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of materials tested and laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency that the 95 % probability limit would imply. The repeatability limit and the reproducibility limit should be considered as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.
- 11.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.
- 11.3 The precision statement was determined through statistical examination of 72 results, from one laboratory, on two materials.
- 11.4 To judge the equivalency of two test results, it is recommended to choose the marking material closest in characteristics to the test material.

³ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1160. Contact ASTM Customer Service at service@astm.org.