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.



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# Standard Test Method for Effect of Moisture on Asphalt Mixtures<sup>1</sup>

This standard is issued under the fixed designation D4867/D4867M; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers procedures for preparing and testing laboratory-compacted asphalt mixture specimens for the purpose of measuring the effect of water on the tensile strength of the paving mixture. This test method is applicable to dense mixtures such as those appearing in the Table for Composition of Bituminous Paving Mixtures in Specification D3515. This test method can be used to evaluate the effect of moisture with or without antistripping additives including liquids and pulverulent solids such as hydrated lime or portland cement.

1.2 The values stated in either SI units or inch-pound units in brackets shall be regarded separately as standard. The values in each system may not be exact equivalents; therefore, each system must be used independently of the other, without combining values in any way.

1.3 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.5 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:<sup>2</sup>
- D8 Terminology Relating to Materials for Roads and Pavements
- D979/D979M Practice for Sampling Asphalt Mixtures
- D1074 Test Method for Compressive Strength of Asphalt Mixtures
- D1561/D1561M Practice for Preparation of Bituminous Mixture Test Specimens by Means of California Kneading Compactor (Withdrawn 2022)<sup>3</sup>
- D2041/D2041M Test Method for Theoretical Maximum Specific Gravity and Density of Asphalt Mixtures
- D2726/D2726M Test Method for Bulk Specific Gravity and Density of Non-Absorptive Compacted Asphalt Mixtures
- D3203 Test Method for Percent Air Voids in Compacted Asphalt Mixtures
- D3387 Test Method for Compaction and Shear Properties of Bituminous Mixtures by Means of the U.S. Corps of Engineers Gyratory Testing Machine (GTM) (Withdrawn 2020)<sup>3</sup>
- D3496 Practice for Preparation of Bituminous Mixture
- Specimens for Dynamic Modulus Testing (Withdrawn 2010)<sup>3</sup>
- D3515 Specification for Hot-Mixed, Hot-Laid Bituminous Paving Mixtures (Withdrawn 2009)<sup>3</sup>
- D3549/D3549M Test Method for Thickness or Height of Compacted Asphalt Mixture Specimens
- D3665 Practice for Random Sampling of Construction Materials
- D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.22 on Effect of Water and Other Elements on Asphalt Coated Aggregates.

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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

- D4013 Practice for Preparation of Test Specimens of Bituminous Mixtures by Means of Gyratory Shear Compactor (Withdrawn 2013)<sup>3</sup>
- D4123 Test Method for Indirect Tension Test for Resilient Modulus of Bituminous Mixtures (Withdrawn 2003)<sup>3</sup>
- D6926 Practice for Preparation of Asphalt Mixture Specimens Using Marshall Apparatus

### 3. Terminology

3.1 Refer to Terminology D8 for definitions relating to materials for roads and pavements.

# 4. Summary of Test Method

4.1 Potential for Moisture Damage—The degree of susceptibility to moisture damage is determined by preparing a set of laboratory-compacted specimens conforming to the job-mix formula without an additive. The specimens are compacted to a void content corresponding to void levels expected in the field, usually in the 6 to 8 % range. The set is divided into two subsets of approximately equal void content. One subset is maintained dry while the other subset is partially saturated with water and moisture conditioned. The tensile strength of each subset is determined by the tensile splitting test. The potential for moisture damage is indicated by the ratio of the tensile strength of the wet subset to that of the dry subset.

4.2 Additive Effect—The effect of an antistripping additive is determined on a set of specimens containing an additive prepared and tested as described in 4.1. The effect of an additive dosage may be estimated by repeating the tests on sets with different additive dosages.

4.3 *Plant-Produced Mixtures*—The potential for moisture damage or the effectiveness of an additive in a plant-produced mixture is determined on specimens that are laboratory compacted to expected field-level void content, divided into wet and dry subsets, and evaluated as described in 4.2.

### 5. Significance and Use

5.1 This test method can be used to test asphalt mixtures in conjunction with mixture design testing to determine the potential for moisture damage, to determine whether or not an antistripping additive is effective, and to determine what dosage of an additive is needed to maximize the effectiveness. This test method can also be used to test mixtures produced in plants to determine the effectiveness of additives under the conditions imposed in the field.

## 6. Apparatus

6.1 To prepare and compact the specimens, use apparatus from any one of the following: Test Methods D1074 and D3387, Practice D3496, or Practices D1561/D1561M, D4013, and D6926.

6.2 *Vacuum Pump or Water Aspirator,* in accordance with Test Method D2041/D2041M.

6.3 *Manometer or Vacuum Gage*, in accordance with Test Method D2041/D2041M.

6.4 *Container*, preferably Type F, of Test Method D2041/ D2041M.

6.5 *Balance*, in accordance with Test Method D2726/ D2726M.

6.6 Water Baths, three:

6.6.1 One water bath in accordance with Test Method D2726/D2726M,

6.6.2 One bath capable of maintaining a temperature of 60  $\pm$  1.0 °C [140  $\pm$  1.8 °F] for 24 h, and

6.6.3 One bath capable of maintaining a temperature of 25  $\pm$  1.0 °C [77  $\pm$  1.8 °F].

6.7 *Mechanical or Hydraulic Testing Machine*, capable of maintaining the required strain rate and measuring load with equal or better precision.

6.8 *Loading Strips*, in accordance with Test Method D4123.

## 7. Preparation of Laboratory Test Specimens

7.1 Make at least six specimens for each test, three to be tested dry and three to be tested after partial saturation and moisture conditioning.

7.2 Use specimens 100 mm [4 in.] in diameter and 62.5 mm [2.5 in.] high, in general, but specimens of other dimensions may be used if desired. When using aggregate larger than 25 mm [1 in.], use specimens at least 150 mm [6 in.] in diameter.

Note 2—The user is cautioned that the specimen diameter has been determined to influence both the tensile strength and the tensile strength ratio. The tensile strength and the tensile strength ratio values may be different for 150 mm specimens compared to 100 mm specimens.

7.3 Prepare mixtures in batches large enough to make at least three specimens or, as an alternative, prepare a batch just large enough for one specimen. If theoretical maximum specific gravity is to be determined, use a batch large enough or prepare a separate batch to provide a specimen for this purpose.

7.4 When a liquid antistripping additive is used, heat a sufficient quantity of asphalt cement for one batch to  $150 \pm 6 \,^{\circ}\text{C} [300 \pm 10 \,^{\circ}\text{F}]$  in a closed 1 L [1 qt] can in an oven. Add the required quantity of additive and immediately mix, for approximately 2 min, with a mechanical stirrer approximately 25 mm [1 in.] from the bottom of the container. Maintain the treated asphalt cement at  $150 \pm 6 \,^{\circ}\text{C} [300 \pm 10 \,^{\circ}\text{F}]$  in the closed can until it is used. Discard the treated asphalt cement if not used the same day it is prepared, or if allowed to cool so that it requires reheating.

Note 1—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Specification D3666 are generally considered capable of competent and objective testing, sampling, inspection, etc. Users of this standard are cautioned that compliance with Specification D3666 alone does not completely ensure reliable results. Reliable results depend on many factors; following the suggestions of Specification D3666 or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.

7.5 When using a pulverulent solid antistripping additive, use the addition procedure simulating the procedure expected in the field. Follow the procedure specified in either 7.5.1, 7.5.2, or 7.5.3.

7.5.1 When dry powder is added to dry aggregate, dry, batch, and heat the mineral aggregate to  $150 \pm 6$  °C [300  $\pm$  10 °F]. Add the required quantity of additive to the aggregate, and thoroughly mix the entire mass until a uniform distribution of additive is achieved. Take care to minimize the loss of additive to the atmosphere in the form of dust. After mixing, maintain the treated aggregate at the required mixing temperature until it is used.

7.5.2 When dry powder is added to damp aggregate, batch the damp mineral aggregate, and adjust the moisture content of the combined aggregate to the expected field moisture level. Add the required quantity of additive to the damp aggregate, and thoroughly mix the entire mass until a uniform distribution of additive is achieved. Take care to minimize the loss of additive to the atmosphere in the form of dust. After mixing, dry the treated aggregate, heat to the required mixing temperature, and maintain at that temperature until it is used.

7.5.3 When powder slurry is used, add the required quantity of additive to water using the powder-to-water ratio expected in the field. Take care to minimize the loss of additive to the atmosphere in the form of dust. To prevent settling, continuously mix the resulting slurry until it is used. Batch the damp mineral aggregate, adjust the moisture content as required in 7.5.2, add the required quantity of slurry, and thoroughly mix the entire mass until a uniform distribution of slurry is achieved. After mixing, dry the treated aggregate, heat to the required mixing temperature, and maintain at that temperature until used.

7.6 Proportion, mix, and compact specimens in accordance with one of the following: Test Methods D1074, D3387, Practice D3496, Practice D1561/D1561M, D4013, or D6926, and 7.6.1 and 7.6.2.

7.6.1 After mixing, stabilize the mixture temperature of each specimen at the required compaction temperature, in a closed container, in an oven for 1 to 2 h. If preparing a multi-specimen batch, split the batch into single-specimen quantities before placing into the oven.

7.6.2 Compact the specimens to  $7 \pm 1$  % air voids, or a void level expected in the field at the time of construction. This void level can be obtained by adjusting the following: the static load in double-plunger compaction; the number of blows in a Marshall hammer compaction; the foot pressure, number of tamps, leveling load, or some combination in kneading compaction; or the number of revolutions in gyratory compaction. Determine the exact procedure by trial for each mixture.

7.6.3 Cool specimens in the mold to room temperature as rapidly as possible in a stream of moving air, extract from molds, then follow the procedure outlined in Section 9 within 24 h.

#### 8. Preparation of Field Specimens

8.1 Select a truck to be sampled in accordance with Practice D3665.

8.2 Secure a sample from the truck at the plant in accordance with Practice D979/D979M.

8.3 Stabilize the mixture temperature to approximately the temperature found in the field when rolling begins. Maintain this temperature in a closed container, in an oven if necessary, for approximately the time lapse between mixing and the start of actual rolling.

8.4 Compact the specimens in accordance with 7.6.2, and cool and extract from the molds in accordance with 7.6.3.

8.5 If specimens are not to be compacted in the field laboratory, place the samples in a sealed container, transport to the laboratory, and reheat to the temperature required in 8.3. Proceed with the steps in 8.4.

Note 3—Specimens made from plant-produced mixtures in accordance with Section 8 may yield different results from specimens made from laboratory-produced mixtures of the same job mix made in accordance with Section 7.

#### 9. Procedure

Note 4—A data sheet that is convenient for use with this procedure appears in Appendix X1.

9.1 Determine the theoretical maximum specific gravity in accordance with Test Method D2041/D2041M.

9.2 Determine the specimen height in accordance with Test Method D3549/D3549M.

9.3 Determine the bulk specific gravity in accordance with Test Method D2726/D2726M, and express the volume of the specimen in cubic centimeters. The term (B-C) in Test Method D2726/D2726M is the volume of the specimen in cubic centimeters.

9.4 Calculate the percent air voids in accordance with Test Method D3203, and express the volume of air in cubic centimeters. The volume of air is the volume of the specimen in 9.3 multiplied by the percent air voids.

9.5 Sort the specimens into two subsets so that the average air voids of the two subsets are approximately equal. Store the subset to be tested dry at room temperature.

9.6 Partially saturate the subset to be moisture conditioned with distilled water at room temperature using a vacuum chamber. If it is difficult to reach the minimum degree of saturation required in 9.6.3, the water used to saturate may be heated up to 60  $^{\circ}$ C [140  $^{\circ}$ F].

9.6.1 Partially saturate, to the degree specified in 9.6.3, by applying a partial vacuum such as 70 kPa or 525 mm Hg [20 in. Hg] for a short time such as 5 min.

Note 5—Experiments with partial vacuum at room temperature indicate that the degree of saturation is very sensitive to the magnitude of the vacuum and practically independent of the duration. The level of vacuum needed appears to be different for different mixtures.

9.6.2 Determine the volume of the partially saturated specimen in accordance with Test Method D2726/D2726M. Determine the volume of the absorbed water by subtracting the air-dry mass of the specimen in 9.3 from the saturated surface-dry mass of the partially saturated specimen.

9.6.3 Determine the degree of saturation by dividing the volume of the absorbed water in 9.6.2 by the volume of air

voids in 9.4 and express the result as a percentage. If the volume of water is between 55 and 80 % of the volume of air, proceed to 9.7. If the volume of water is less than 55 %, repeat the procedure beginning with 9.6.1 using a slightly higher partial vacuum. If the volume of water is more than 80 %, the specimen has been damaged and is discarded.

Note 6—If the average air voids of the saturated subset is less than 6.5 %, a degree of saturation of at least 70 % is recommended.

9.7 Moisture condition the partially saturated specimens by soaking in distilled water at 60  $\pm$  1.0 °C [140  $\pm$  1.8 °F] for 24 h.

Note 7—If a freeze-thaw conditioning cycle is desired, the following procedure is suggested instead of the procedure in 9.7. Wrap each of the partially saturated specimens tightly with two layers of plastic film, using masking tape to hold the wrapping if necessary. Place each wrapped specimen into a leak-proof plastic bag containing approximately 3 mL of distilled water, and seal the bag with a tie or tape. Place the wrapped and bagged specimens into an air bath freezer at  $-18 \pm 2.0$  °C [ $-0.4 \pm 3.6$  °F]. After at least 15 h in the freezer, remove the specimens and immerse them in a water bath at 60  $\pm$  1.0 °C [ $140 \pm 1.8$  °F] for 24 h. After 3 min of immersion, after specimen surface thaw occurs, remove the bag and wrapping from the specimens.

9.8 Adjust the temperature of the moisture-conditioned subset by soaking in a water bath for 1 h at 25  $\pm$  1 °C [77  $\pm$  1.8 °F].

9.9 Measure the height of the moisture-conditioned subset by Test Method D3549/D3549M, and determine volume by Test Method D2726/D2726M.

9.9.1 Determine the water absorption and the degree of saturation in accordance with 9.6.2 and 9.6.3. A degree of saturation exceeding 80 % is acceptable.

9.9.2 Determine the swell of the partially saturated specimens by dividing the change in specimen volumes in 9.6.2 and 9.3 by the specimen volume in 9.3. Determine the swell of moisture-conditioned specimens by dividing the change in the specimen volume in 9.9 and 9.3 by the specimen volume in 9.3.

9.10 Adjust the temperature of the dry subset by soaking in a water bath for 20 min at 25  $\pm$  1.0 °C [77  $\pm$  1.8 °F].

9.11 Determine the tensile strength at 25  $\pm$  1.0 °C [77  $\pm$  1.8 °F] of both subsets.

9.11.1 Place a specimen into the loading apparatus and position the loading strips so that they are parallel and centered on the vertical diametral plane. Apply a diametral load at 50 mm/min [2 in./min] until the maximum load is reached, and record the maximum load.

9.11.2 Continue loading until the specimen fractures. Break the specimen open and visually estimate and record the approximate degree of moisture damage, if any.

9.11.3 Inspect all surfaces, including the failed faces, for evidence of cracked or broken aggregate, that may influence test results, and record observations.

#### 10. Calculation

10.1 Calculate the tensile strength as follows:

$$S_{t} = 2000 P \pi t D (kPa)$$
(1)

$$S_t = 2P/\pi t D$$
 (psi

where:

 $S_{\rm t}$  = tensile strength, kPa [psi],

P = maximum load, N [lbf],

*t* = specimen height immediately before tensile test, mm [in.], and

D = specimen diameter, mm [in.].

10.2 Calculate the tensile strength ratio as follows:

$$TSR = (S_{\rm tm}/S_{\rm td})100 \tag{2}$$

where:

TSR = tensile strength ratio, %,

 $S_{\rm tm}$  = average tensile strength of the moisture-conditioned subset, kPa [psi], and

 $S_{td}$  = average tensile strength of the dry subset, kPa [psi].

#### 11. Report

11.1 Report the following information:

11.1.1 Number of specimens in each subset,

11.1.2 Average air voids of each subset,

11.1.3 Average degree of saturation after partial saturation and after moisture conditioning,

11.1.4 Average swell after partial saturation and after moisture conditioning,

11.1.5 Tensile strength of each specimen in each subset,

11.1.6 Tensile strength ratio,

11.1.7 Results of visually estimated moisture damage observed when the specimen fractures, and

11.1.8 Results of observations of fractured or crushed aggregate.

Note 8—If the conditioning procedure described in Note 7 is used, that fact should be included in the report.

# 12. Precision and Bias 213 d/astm-d4867-d4867m-22

12.1 *Precision*—The standard deviations for use with this test method have been determined using laboratory-mixed specimens conditioned in accordance with 9.7. Neither plant-mixed material nor the conditioning in Note 7 has been studied. Nineteen laboratories participated in the precision study by testing five asphalt mixtures, two of which contained a liquid antistripping additive.

12.1.1 *Within-Laboratory Precision*—The single-operator standard deviation of tensile strength for either dry or moisture-conditioned specimens has been found to be 55 kPa [8 psi]. The d2s limit for the maximum allowable difference in tensile strength between duplicate specimens of the same mixture tested by the same operator is 159 kPa [23 psi].

12.1.2 *Between-Laboratory Precision*—The multilaboratory standard deviation of the tensile strength ratio has been found to be 8 %. The d2s limit for the maximum allowable difference in tensile strength ratio between results of tests performed on samples of the same mixture by two different laboratories is 23 %.

12.2 *Bias*—This test method has an undetermined bias because the value of a tensile strength ratio can be defined only in terms of the test method.