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.

Designation: D1754/D1754M – 20

Standard Test Method for Effects of Heat and Air on Asphaltic Materials (Thin-Film Oven Test)[1]

This standard is issued under the fixed designation D1754/D1754M; 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 the determination of the effects of heat and air on a film of semisolid asphaltic materials. The effects of this treatment are determined from measurements of selected asphalt properties before and after the test.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 **Warning—**Mercury has been designated by the United States Environmental Protection Agency and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/index.htm—for additional information. Users should be aware that selling mercury and/or mercury-containing products into your state may be prohibited by state law.

1.4 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.

1.5 *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]

D5/D5M Test Method for Penetration of Bituminous Materials

D8 Terminology Relating to Materials for Roads and Pavements

D2170/D2170M Test Method for Kinematic Viscosity of Asphalts

D2171/D2171M Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer

D3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D8055 Guide for Selecting an Appropriate Electronic Thermometer for Replacing Mercury Thermometers in D04 Road and Paving Standards

E1 Specification for ASTM Liquid-in-Glass Thermometers

E77 Test Method for Inspection and Verification of Thermometers

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature

E644 Test Methods for Testing Industrial Resistance Thermometers

3. Terminology

3.1 Definitions of terms used in this test method may be found in Terminology D8, determined from common English usage, or combinations of both.

[1] This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.46 on Durability and Distillation Tests.

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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.

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4. Summary of Test Method

4.1 A film of asphaltic material is heated in an oven for 5 h at 163 °C [325 °F]. The effects of heat and air are determined from changes incurred in physical properties measured before and after the oven treatment. An optional procedure is provided for determining the change in sample mass.

4.2 Precision values for the method have been developed for viscosity, viscosity change, penetration change, mass change, and ductility.

5. Significance and Use

5.1 This method indicates approximate change in properties of asphalt during conventional hot-mixing at about 150 °C [302 °F] as indicated by viscosity, penetration, or ductility measurements. It yields a residue which approximates the asphalt condition as incorporated in the pavement. If the mixing temperature differs appreciably from the 150 °C [302 °F] level, more or less effect on properties will occur.

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.

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6. Apparatus

6.1 *Oven*—The oven shall be electrically heated and shall conform to the performance requirements of Specification E145, Type IB (Gravity-Convection), for operating temperatures up to 180 °C [356 °F]. During the tests for compliance to Specification E145 requirements, the oven shelf, properly centered as described in 6.1.2, shall be in place and rotating.

ASTM D1754/D1754M-20

6.1.1 *Construction*—The oven shall be rectangular, and each interior dimension (exclusive of space occupied by the heating element) shall be a minimum of 330 mm [13 in.] and a maximum of 535 mm [21 in.]. The oven shall have, in front, a tightly fitted hinged door, which shall provide a clear opening substantially the same as the interior height and width of the oven. The door may contain a window with dimensions of at least 100 by 100 mm [4 by 4 in.] and with two sheets of glass separated by an air space, through which a vertical thermometer, located as specified in 6.2, may be read without opening the door; or the oven may be provided with an inner glass door, through which the thermometer may be observed on opening the outer door momentarily. The oven shall be adequately ventilated by convection currents of air and for this purpose shall be provided with openings for the entrance of air and for the exit of heated air and vapors. Openings may be of any size and arrangement provided the requirements of Specification E145, Type IB, are met.

6.1.2 *Rotating Shelf*—The oven shall be provided with a single metal circular shelf having a minimum diameter of 250 mm [9.8 in.] and a maximum diameter of 450 mm [18 in.]. The shelf construction shall be such that it provides a flat surface for support of the containers without blocking all air circulation through the shelf when the containers are in place. The shelf shall be suspended by a vertical shaft and centered with respect to the horizontal interior dimensions of the oven and shall be provided with a mechanical means of rotating it at the rate of 5.5 ± 1.0 r/min. The preferred vertical position for the shelf is 150 mm [6 in.] above the bottom of the oven (exclusive of space occupied by the heating element), and the shelf shall be located as close to this position as permitted by compliance with the requirements of 6.2 regarding thermometer placement. The shelf shall be constructed or marked in such a way that the sample containers can be placed in the same position during each test. There shall be a minimum of two and a maximum of six sample container positions. Each sample container position shall be symmetrical with respect to the shaft and to any holes in the shelf. The number of sample container positions shall be the maximum that will fit on the shelf without violating the above requirements and without excessive overhang.

6.2 *Thermometer*—A thermometer for measuring temperature in the oven. The thermometer shall be one of the following (see Note 2):

6.2.1 A liquid-in-glass thermometer which conforms to the requirements of Specification E1 readable to 0.5 °C [1 °F] that includes the test temperature in its calibrated or standardized range, 163 °C [325 °F]. Standardize the thermometer in accordance with one of the methods in Test Method E77 or verify its original standardization at the ice point in accordance with Practice E563. If the thermometer does not read to 0 ± 0.5 °C [32 ± 1 °F] at the ice point, then the thermometer should be re-standardized.

6.2.2 A platinum resistance thermometer (PRT) readable to the nearest 0.5 °C [1 °F], with a PT 100 Class AA tolerance rating and either a three- or four-wire configuration and an overall sheath length at least 50 mm [2 in.] greater than the immersion depth. The thermometer shall have the test temperature, 163 °C [325 °F], within its calibrated or standardized range. Standardize the PRT system (probe and readout device) in accordance with Test Method E644. Corrections shall be applied to ensure accurate measurements within 0.5 °C [1 °F].

NOTE 2—Guide D8055 provides additional guidance on selecting appropriate electronic thermometer alternatives to a mercury-in-glass thermometer.

6.3 *Container*—Cylindrical pans, 140 ± 1 mm [5.5 ± 0.04 in.] in inside diameter and 9.5 ± 1.5 mm [3⁄8 ± 1⁄16 in.] deep with a flat bottom. Fifty milliliters of the sample in this size container give a film thickness of approximately 3.2 mm [1⁄8 in.]. Pans shall be made of stainless steel and shall have a metal thickness of approximately 0.64 mm [0.025 in.].

NOTE 3—Pans have a tendency to become warped or bent with use. Although tests indicate that a small amount of warping does not significantly affect results, frequent inspection to eliminate warped or damaged pans is advisable. The indicated metal thickness has been found to provide adequate rigidity without excessive weight. Stainless steel pans manufactured from 0.6-mm thick [No. 24] stainless sheet gauge steel comply with the recommended thickness. Pans made from 0.48 mm-thick [No. 26] stainless sheet gauge metal are also acceptable but have a greater tendency to warp during use.

6.4 *Balance*—A balance conforming to the requirements of Guide D4753, Class G2.

7. Preparation of Oven

7.1 Ensure that the thermometric device tip is positioned 40 mm [1.5 in.] above the top of the shelf and centered over the arc of the rotating pans.

7.2 Ensure the oven is level so that the shelf rotates in a horizontal plane. The maximum tilt during rotation shall not be more than 3° from the horizontal.

7.3 Preheat oven for a minimum of 2 h prior to testing, with the control setting adjusted to the setting that will be used during the test. The setting shall be selected such that when the oven is fully loaded, the oven will equilibrate at 163.0 ± 1 °C [325 ± 2 °F].

NOTE 4—Removing sample containers will affect convection patterns in the oven, and may cause the temperature reading to change from the desired reading. This is normal, and occurs because the thermometric device is not in the same location as the temperature control sensor. Replacing the containers should cause the temperature reading to return to the original reading.

8. Preparation of Samples

8.1 Place sufficient material for the test in a suitable container and heat to a fluid condition. Extreme care should be taken so that there is no local overheating of the sample and that the highest temperature reached is not more than 163 °C [325 °F]. Stir the sample during the heating period, but avoid incorporating air bubbles in the sample. Transfer 50 ± 0.5 g into each of two or more tared containers meeting the requirements of 6.3.

8.2 At the same time, pour a portion of the sample into the containers specified for measurement of original asphalt properties. Complete the tests by appropriate ASTM Test Method D5/D5M, D2170/D2170M, or D2171/D2171M.

8.3 If the quantitative value of the mass change is desired, cool the samples for the oven test to room temperature and determine the mass of each sample separately to the nearest 0.001 g. If the mass change is not required, allow the samples to cool to approximately room temperature before placing in the oven as directed in 9.1.

9. Procedure

9.1 With the oven preheated and adjusted as described in 7.3, quickly place the asphalt samples in the predetermined sample container positions on the circular shelf (Note 5 and Note 6). Fill any vacant positions with empty sample containers, so that every sample container position is occupied. Close the oven door and start rotating the shelf. Maintain the specified temperature range for 5 h after the sample has been introduced and the oven has again reached that temperature. The 5-h period shall start when the temperature reaches 162 °C [323 °F] and in no case shall the total time that a sample is in the oven be more than 5¼ h. At the conclusion of the heating period, remove the samples from the oven. If the mass change is not being determined, proceed in accordance with 9.3. If the mass change is being determined, cool to room temperature, determine the mass to the nearest 0.001 g, and calculate the mass change on the basis of the asphalt in each container (Note 7).

NOTE 5—Materials having different mass change characteristics should not generally be tested at the same time due to the possibility of cross-absorption.

NOTE 6—This test method does not prohibit placing an asphalt sample in the position under the mercury thermometer. However, it is recommended that this position not be used for a sample, and that an empty pan remain in this position, in order to minimize the risk associated with thermometer breakage.

NOTE 7—When complete tests cannot be made in the same day, and if the mass change is being determined, determine the masses of the residues and store them overnight before reheating. If the mass change is not being determined, transfer the residue to the 240-mL [8-oz] container as described in 9.3 before storing overnight.

9.2 After determining the mass of the samples, place them on a refractory-board and then on the shelf of the oven maintained at 163 °C [325 °F]. Close the oven and rotate the shelf for 15 min and immediately proceed as described in 9.3.

9.3 Remove each pan individually and transfer the contents by pouring and scraping into a seamless metal container of approximately 240-mL [8-oz] capacity. Remove substantially all of the material from the pans by scraping with a suitable tool such as a spatula or putty knife. While the residue is being removed from each pan, the oven door shall remain closed, with the heater power on, and the remaining samples rotating on the shelf. The final pan shall be removed from the oven within 5 min of the removal of the initial pan. Stir the combined residues thoroughly, in a semi-fluid state, until homogenous. If needed, heat the combined residues in the covered metal container in an oven at a temperature not exceeding 163 °C [325 °F]. Complete the tests on residue by appropriate ASTM Test Method D5/D5M, D2170/D2170M, or D2171/D2171M within 72 h of performing this test.

NOTE 8—Care should be taken if the sample is reheated, since excessive reheating can affect the apparent severity of the test.

10. Report

10.1 Report the values of the original asphalt properties measured in 8.2 and the residue property values as measured in 9.3. Viscosity change may also be expressed as the ratio of the residual asphalt viscosity to the original asphalt viscosity. Penetration change is evaluated as the penetration of the residue expressed as the percentage of the original penetration.

10.2 Report ductility or other test results in accordance with the appropriate ASTM test methods.

10.3 When determined, report the average mass change of the material in all containers as mass percent of the original material. A mass loss shall be reported as a negative number, while a mass gain shall be reported as a positive number.

NOTE 9—This test can result in either a mass loss or a mass gain. During the test, volatile components evaporate (causing a decrease in mass), while oxygen reacts with the sample (causing an increase in mass). The combined effect determines whether the sample has an overall mass gain or an overall mass loss. Samples with a very low percentage of volatile components will usually exhibit a mass gain, while samples with a high percentage of volatile components will usually exhibit a mass loss.

NOTE 10—If a skin formation is noted during testing, it should be included in the report.

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11. Precision and Bias

11.1 Criteria for judging the acceptability of the viscosity at 60 °C [140 °F] and 135 °C [275 °F], viscosity ratio at 60 °C [140 °F], change in penetration at 25 °C [77 °F], ductility at 15.6 °C [60 °F] (average of three measurements), and mass change test results obtained by this method are given in Table 1. The figures given in Column 2 are the standard deviations that have been found to be appropriate for unmodified materials and the conditions of test described in Column 1. The figures given in Column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests. The figures given in Column 4 are the coefficients of variation that have been found to be appropriate for the materials and conditions of test described in Column 1. The figures given in Column 5 are the limits that should not be exceeded by the difference between the results of two properly conducted tests expressed as a percent of their mean.

11.2 *Bias*—The bias for this test method cannot be determined because no material with an accepted reference value is available.

12. Keywords

12.1 aging; asphalt cement; thin-film oven test (TFOT)

TABLE 1 Precision of Test on TFOT Residue

Material and Type Index	Standard Deviation (1s)	Acceptable Range of Two Results (d2s)	Coefficient of Variation (percent of mean) (1s%)	Acceptable Range of Two Results (percent of mean) (d2s%)
Single-operator precision:				
Percentage of retained penetration	1.43	4.0	...	...
Change in mass percentage:				
Not more than 0.4 % (max)	0.014	0.04	...	...
Greater than 0.4 %	...	...	2.9	8.0
Viscosity at 60 °C [140 °F]	...	...	3.3	9.3
Viscosity at 135 °C [275 °F]	...	...	2.0	5.7
Ratio: $\frac{\text{viscosity at 60 °C [140 °F] after test}}{\text{viscosity at 60 °C [140 °F] before test}}$	...	...	5.6	16.0
Ductility at 15.6 °C [60 °F], cm[A]	7	20		
Multilaboratory precision:				
Percentage of retained penetration	2.90	8.0	...	...
Change in mass percentage:				
Not more than 0.4 % (max)	0.055	0.16	...	...
Greater than 0.4 %	...	...	14.0	40.0
Viscosity at 60 °C [140 °F]	...	...	11.6	33.0
Viscosity at 135 °C [275 °F]	...	...	6.4	18.0
Ratio:[B] $\frac{\text{viscosity at 60 °C [140 °F] after test}}{\text{viscosity at 60 °C [140 °F] before test}}$	...	...	9.1	26.0
Ductility at 15.6 °C [60 °F], cm[A]	12	34		

[A] This is based on the analysis of data resulting from tests by 60 laboratories on four asphalts with average ductilities ranging from 20 to 40 cm.
[B] Multilaboratory precision applicable to asphalt cements having viscosity ratios lower than 3.0. Precision for ratios greater than 3.0 have not been established.

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