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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 D 1754; 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 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 5 Test Method for Penetration of Bituminous Materials[2]

D 113 Test Method for Ductility of Bituminous Materials[2]

D 2170 Test Method for Kinematic Viscosity of Asphalts (Bitumens)[2]

D 2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer[2]

E 1 Specification for ASTM Thermometers[3]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[4]

3. Summary of Test Method

3.1 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.

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

4. Significance and Use

4.1 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.

5. Apparatus

5.1 *Oven*—The oven shall be electrically heated and shall conform to the performance requirements of Specification E 145, Type IB (Gravity-Convection), for operating temperatures up to 180°C (356°F). During the tests for compliance to Specification E 145 requirements, the oven shelf, properly centered as described in 5.1.2 shall be in place and rotating.

5.1.1 *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 5.2, 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 E 145, Type IB, are met.

5.1.2 *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

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[2] *Annual Book of ASTM Standards*, Vol 04.03.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] *Annual Book of ASTM Standards*, Vol 14.04.

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 5.2 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.

5.2 *Thermometer*—An ASTM Loss on Heat Thermometer having a range from 155 to 170°C and conforming to the requirements for Thermometer 13C, as prescribed in Specification E 1 shall be used for determining the test temperature. The thermometer shall be supported from the shaft of the circular shelf in a vertical position at a point equidistant from the center and outer edge of the shelf. The bottom of the thermometer bulb shall be 40 mm (1.5 in.) above the top of the shelf. The thermometer shall be radially centered over a sample container position..

5.3 *Container*—A cylindrical pan, 140 mm (5½ in.) in inside diameter and 9.5 mm (⅜ in.) deep with a flat bottom. Fifty millilitres of the sample in this size container give a film thickness of approximately 3.2 mm (⅛ in.). Pans shall be made of stainless steel and shall have a metal thickness of approximately 0.64 mm (0.025 in.).

NOTE 1—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 gage steel comply with the recommended thickness. Pans made from 0.48 mm-thick (No. 26) stainless sheet gage metal are also acceptable but have a greater tendency to warp during use.

6. Preparation of Samples

6.1 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 150°C (302°F). Stir the sample during the heating period, but avoid incorporating air bubbles in the sample. Weigh 50± 0.5 g into each of two or more tared containers meeting the requirements of 5.3.

6.2 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 methods.

6.3 If the quantitative value of the mass change is desired, cool the samples for the oven test to room temperature and weigh 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 7.2.

7. Procedure

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

7.2 Place an empty sample container in each of the predetermined sample container positions on the rotating shelf. Adjust the temperature control so the specified thermometer (see 5.2) reads 163 ± 1°C (325 ± 2°F) when the oven is at equilibrium. Once adjustment is complete, the empty sample containers may be removed at the discretion of the operator. However, the temperature control shall not be readjusted once any sample containers are removed.

NOTE 2—Removing sample containers will affect convection patterns in the oven, and may cause the thermometer reading to change from the desired level. This is normal, and occurs because the thermometer is not in the same location as the temperature control sensor. Replacing the containers should cause the thermometer reading to return to the original level.

7.3 With the oven preheated and adjusted as described in 7.2, quickly place the asphalt samples in the predetermined sample container positions on the circular shelf (Note 3 and Note 4). 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 7.5. If the mass change is being determined, cool to room temperature, weigh to the nearest 0.001 g, and calculate the mass change on the basis of the asphalt in each container (Note 5).

NOTE 3—Materials having different mass change characteristics should not generally be tested at the same time due to the possibility of cross-absorption.

NOTE 4—This test method does not prohibit placing an asphalt sample in the position under the 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 5—When complete tests cannot be made in the same day, and if the mass change is being determined, weigh 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 7.5 before storing overnight.

7.4 After weighing the samples, place them on a refactory-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, remove the samples and board(s), and immediately proceed as described in 7.5.

7.5 Transfer the material from each pan into an 240-mL (8-oz) ointment tin. Remove substantially all of the material from the pans by scraping with a suitable spatula or putty knife. Stir the combined residues thoroughly, placing the 240-mL (8-oz) container on a hot plate to maintain the material in a

fluid condition if necessary. Complete the tests on residue by appropriate ASTM test methods within 72 h of performing this test.

NOTE 6—Care should be taken if the sample is reheated more than once, since excessive reheating can affect the apparent severity of the test.

8. Report

8.1 Report the values of the original asphalt properties measured in 6.2 and the residue property values as measured in 7.5. 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.

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

8.3 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 7—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.

9. Precision and Bias

9.1 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), 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 the materials and 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.

9.2 Criteria for judging the acceptability of ductility data at 15.6°C (60°F) are given in Table 1. Each test result is the average of three ductility measurements.

10. Keywords

10.1 aging; asphalt cement; Thin-Film Oven Test (TFOT)

TABLE 1 Precision of Test on 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: $\frac{\text{viscosity at } 60°C\ (140°F) \text{ after test}}{\text{viscosity at } 60°C\ (140°F) \text{ before test}}$	...	...	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] $\frac{\text{viscosity at } 60°C\ (140°F) \text{ after test}}{\text{viscosity at } 60°C\ (140°F) \text{ before test}}$	...	...	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.