Designation: D2872 – 12ε1

Standard Test Method for Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)[1]

This standard is issued under the fixed designation D2872; 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.

ε^1 NOTE—This standard was corrected editorially in February 2013.

1. Scope

1.1 This test method is intended to measure the effect of heat and air on a moving film of semi-solid asphaltic materials. The effects of this treatment are determined from measurements of the selected properties of the asphalt before and after the test.

1.2 The values stated in inch-pound 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:*[2]

D113 Test Method for Ductility of Bituminous Materials
D2171 Test Method for Viscosity of Asphalts by Vacuum Capillary Viscometer
E1 Specification for ASTM Liquid-in-Glass Thermometers
E644 Test Methods for Testing Industrial Resistance Thermometers
E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

3. Summary of Test Method

3.1 A moving film of asphaltic material is heated in an oven for 85 min at 325°F (163°C). The effects of heat and air are determined from changes in physical test values as measured before and after the oven treatment. An optional procedure is provided for determining the change in sample mass.

3.2 Precision values for this test method have been developed for viscosity at 140°F (60°C); ductility at 60°F (15.6°C); and mass change.

4. Significance and Use

4.1 This test method indicates approximate change in properties of asphalt during conventional hot-mixing at about 302°F (150°C) as indicated by viscosity and other rheological measurements. It yields a residue which approximates the asphalt condition as incorporated in the pavement. If the mixing temperature differs appreciably from the 302°F (150°C) level, more or less effect on properties will occur. This test method also can be used to determine mass change, which is a measure of asphalt volatility.

5. Apparatus

5.1 *Oven*—This shall be a double-walled electrically heated convection-type oven. Its inside dimensions shall be 15 in. (381 mm) high, 19 in. (483 mm) wide (including the plenum), and 17½ ± ½ in. (445 ± 13 mm) deep (with the door closed). The door shall contain a symmetrically located window with dimensions of 12 to 13 in. (305 to 330 mm) wide by 8 to 9 in. (203 to 229 mm) high. The window shall contain two sheets of heat-resistant glass separated by an air space. The window should permit an unobstructed view of the interior of the oven. The top of the upper heating element shall be 1 ± ⅛ in. (25 ± 3 mm) below the oven floor.

5.1.1 The oven shall be vented at the top and bottom. The bottom vents shall be located symmetrically to supply incoming air around the heating elements. They shall have an open area of 2.31 ± 0.11 $in.^2$ (15.0 ± 0.7 cm^2). The top vents shall be symmetrically arranged in the upper part of the oven and have an open area of 1.45 ± 0.07 $in.^2$ (9.3 ± 0.45 cm^2).

5.1.2 The oven shall have an air plenum covering the side walls and ceiling. The air space shall be 1½ in. (38.1 mm) deep from the walls and ceiling. At a midpoint in the width of the oven, and 6 in. (152.4 mm) from the face of the circular metal carriage to its axis, a squirrel cage-type fan 5¼ in. (133 mm) OD by 2⅞ in. (73 mm) wide shall be turned at 1725 rpm by an externally mounted motor. The squirrel cage fan shall be set so that the fan turns in an opposite direction to its vanes. The air

[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.

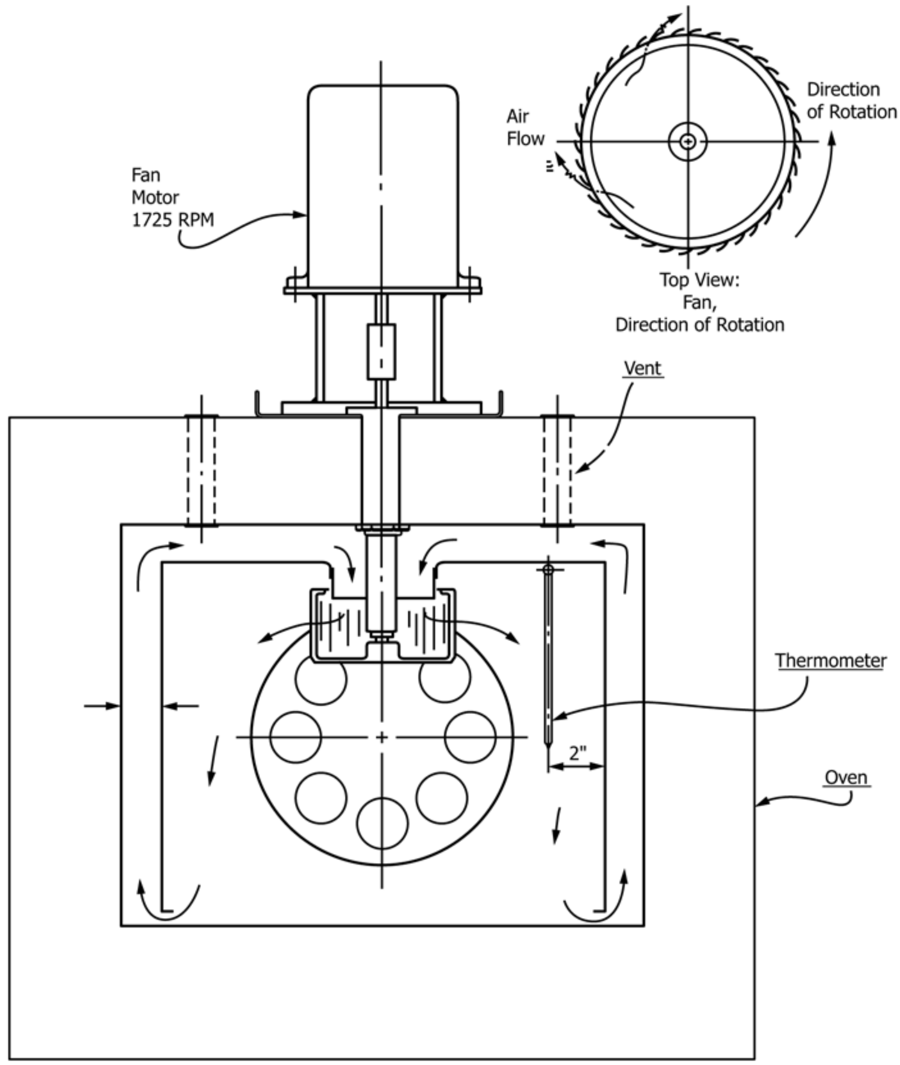

FIG. 1 Schematic of Air Flow Front View

flow characteristics of the fan-plenum system shall be suction from the floor of the oven through the wall plenums and exhaust of the air through the fan. Fig. 1 and Fig. 2 show details of this plenum system.

5.1.3 The oven shall be equipped with a proportional control thermostat capable of maintaining a temperature of 325°F (163°C) to within ±1.0°F (±0.5°C). The sensing element of the thermostat may be placed at any location that enables the oven to maintain temperature control as specified by this standard.

5.1.4 The thermometer shall be hung or affixed to a mounting in the ceiling which is 2 in. (50.8 mm) from the right side of the oven at a midpoint in the depth of the oven. The thermometer shall hang down into the oven so that the bulb of the thermometer is within 1 in. of an imaginary line level with the shaft of the circular metal carriage. The heating controls shall be capable of bringing the fully loaded oven back to the test temperature within a 10-min period after insertion of the samples in a preheated oven.

5.1.5 The oven shall be provided with a 12-in. (304.8-mm) diameter, vertical circular carriage (see Fig. 2 for details). This carriage shall be provided with suitable openings and clips for firmly holding eight glass containers in a horizontal position (see Fig. 3). The vertical carriage shall be mechanically driven through a ¾-in. (19-mm) diameter shaft at a speed of 15 ± 0.2 r/min.

5.1.6 The oven shall be equipped with an air jet positioned to blow heated air into each bottle at its lowest point of travel. The air jet shall have an outlet orifice 0.04 in. (1.016 mm) in diameter (No. 60 drill) connected to a 25-ft (7.6-m) length of 5⁄16-in. (8-mm) outside diameter refrigeration copper tubing. This tubing shall be coiled to lie flat on the bottom of the oven and lead to a source of fresh, dried, dust-free regulated air.

NOTE 1—Activated silica gel treated with an indicator is a satisfactory desiccant for the dried air.

5.2 *Flowmeter*—The flowmeter may be any suitable type capable of accurately measuring the airflow at a rate of 4000 mL/min. The flowmeter shall be located downstream of all regulating devices and upstream of the copper coil. The flowmeter shall be positioned so it is maintained at approximately room temperature. The airflow shall be calibrated

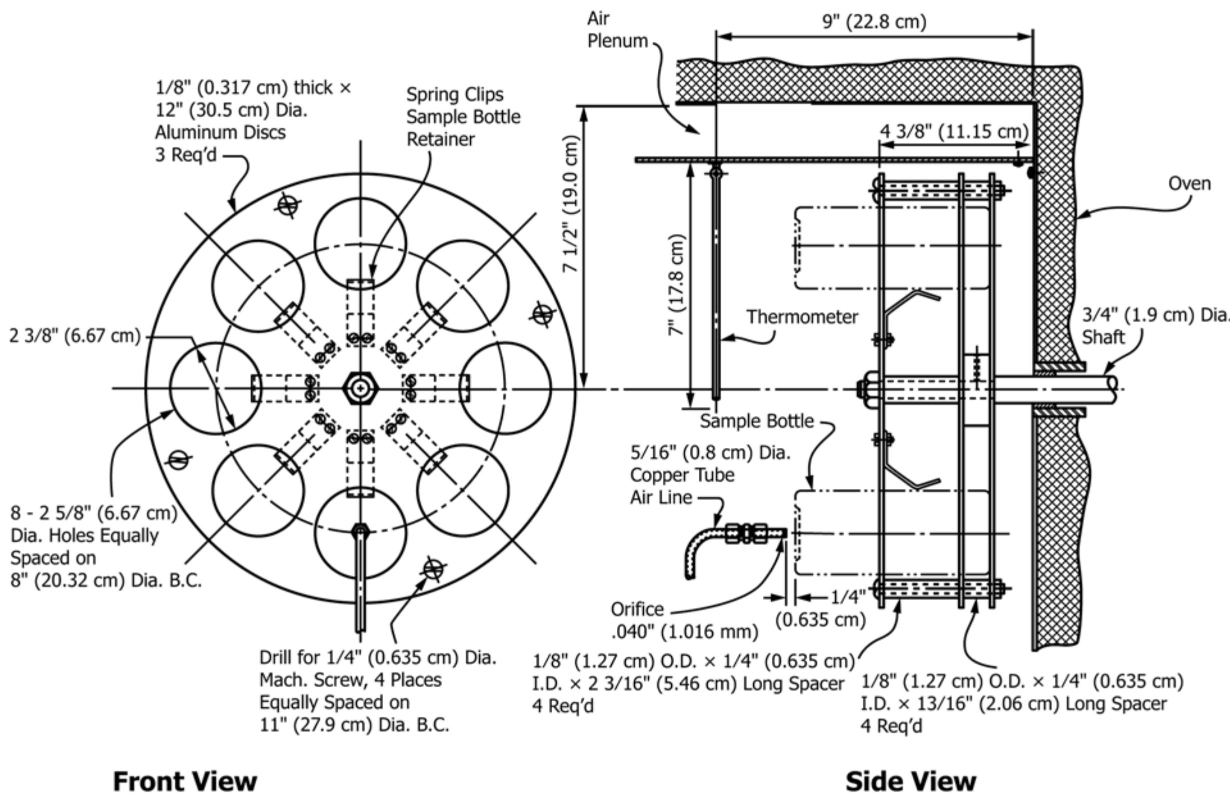

FIG. 2 Circular Metal Carriage

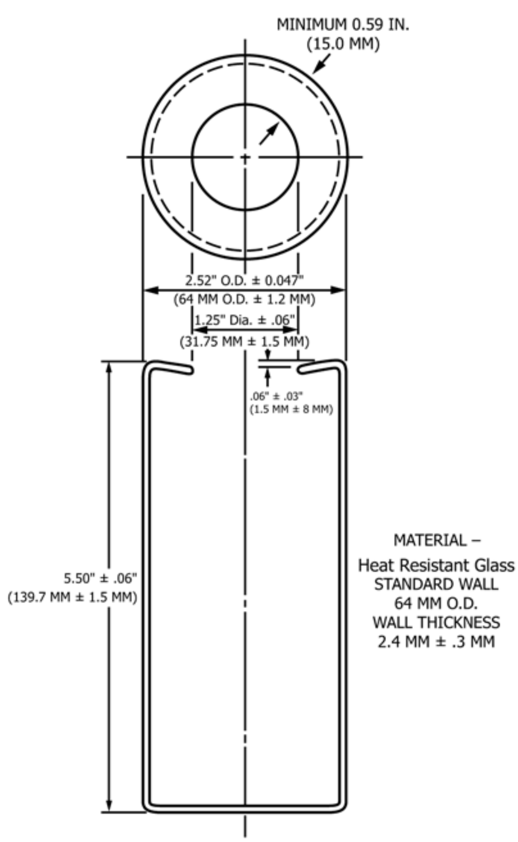

FIG. 3 Sample Bottle

periodically using a wet-test meter or other displacement method. This calibration shall be based on airflow exiting the air jet and shall be conducted with the oven off and at room temperature.

5.3 *Thermometer*—This shall be an ASTM Thermometer conforming to the requirements for Thermometer 13C as prescribed in Specification E1. This thermometer shall be used to make all temperature measurements required by this test method. In order to reduce the risks associated with thermometer breakage, the thermometer may be fully or partially encapsulated in an optically transparent polymer sheath having a maximum thickness of 0.01 in. (0.25 mm). If a sheath is used, it shall be installed such that there is substantial mechanical contact with the thermometer. The thermometer shall be recalibrated after installation of a sheath.

5.3.1 The test thermometer may be replaced with an electronic temperature measurement system, provided the following requirements are met:

5.3.1.1 The electronic temperature sensor shall be a 3 or 4 wire, Grade A Platinum Resistance Thermometer (PRT) substantially conforming to the requirements of Specification E1137/E1137M. The temperature sensor shall be mounted in the same position and orientation as the test thermometer it replaces.

5.3.1.2 The electronic sensor shall have a thermal response time that differs by no more than 30 % from the thermal response time of the designated test thermometer. Thermal response shall be defined as the time required to achieve a 95 % response to a temperature step change, starting in air at ambient temperature, and ending in air at any convenient and constant temperature in the range of 165–170°C. Guidance for determining thermal response time is given in Test Methods E644.

5.3.1.3 The electronic measurement circuitry shall include a digital display having a resolution of 0.1°C or better.

5.3.1.4 The electronic temperature sensor and electronic measurement circuitry shall be calibrated and verified as a unit. Calibration and verification shall be NIST traceable. The temperature measurement system shall be calibrated prior to being placed into service and verified annually thereafter. Guidance for performing the calibration is given in Test Methods E644.

5.3.1.5 Verification shall be conducted with the test oven equilibrated at normal operating temperature while the verification sensor is in mechanical contact with the normal test sensor. If the difference between the verification sensor and the normal test sensor exceeds 0.2°C, the verification will be regarded as having failed, and the temperature measurement system shall be recalibrated.

5.4 *Container*—The container in which the sample is to be tested shall be of clear, transparent, heat-resistant glass conforming to the dimensions shown in Fig. 3.

5.5 *Cooling Rack*—A wire or sheet metal rack, constructed of stainless steel or aluminum, which allows the sample containers to cool in a horizontal position, with each container in the same horizontal plane. The rack shall be constructed in a way that allows air to flow freely around each container with at least 1 in. (2.5 cm) clearance between containers and at least 1 in. (2.5 cm) clearance between the containers and any solid surface.

6. Determination of Oven Preheat Time

6.1 Determine the preheat time for the oven in accordance with either 6.1.1 or 6.1.2. If Section 6.1.1 is used, this determination must be made for each oven, and shall be repeated at least annually or whenever environmental conditions or the test location change. If Section 6.1.2 is used, no annual determination is necessary.

6.1.1 Adjust the oven control thermostat to the setting that will be used during the test. Select this setting so that when the oven is fully loaded and the air is on, the oven will equilibrate at 325 ± 1°F (163 ± 0.5°C), as indicated by the test thermometer. Turn the oven on and simultaneously record the start time to the nearest whole minute. Determine and record the temperature of the oven at 15-minute intervals. Continue this process until the oven reaches thermal equilibrium. Thermal equilibrium is considered to be the time when the oven temperature does not vary by more than 1ºF (0.5ºC) between two consecutive readings. The oven preheat time is the time that it takes to reach thermal equilibrium plus an additional 30 minutes.

6.1.2 In lieu of completing the steps described in Section 6.1.1, a minimum preheat time of 4 hours may be used.

7. Preparation of Oven

7.1 Position the air outlet orifice so that it is ¼ in. ± ⅛ in. (6 mm ± 3 mm) from the opening of the glass container. The orifice shall also be so positioned that the jet blows horizontally into the central arc of the opening of the circling glass container.

7.2 Position the thermometer specified in 5.3 so that the end of the bulb of the thermometer is within 1 in. (25.4 mm) of an imaginary line level with the center of the shaft holding the revolving carriage.

7.3 Level the oven so that the horizontal axes of the glass containers when in position in the carriage are level to within ± 1.0°.

7.4 Start the fan. The fan shall remain on whenever the oven heater is on and the oven door is closed. This standard permits (but does not require) the fan to be stopped when the oven door is opened. Stopping the fan may be accomplished manually, with an electronic door interlock, or through other means.

7.5 Preheat the oven for the preheat time determined in 6 or longer prior to testing with the control thermostat adjusted to the setting that will be used during the test. Select this setting so that when the oven is fully loaded and the air is on, the oven will equilibrate at 325 ± 1°F (163 ± 0.5°C), as indicated by the test thermometer.

NOTE 2—Because the presence of sample containers affects the temperature distribution in the oven, containers should be present in the oven when the thermostat setting is determined. The use of empty containers is acceptable for this purpose.

8. Procedure

8.1 The sample as received shall be free of water. Heat the sample in its container with a loosely fitted cover in an oven not to exceed 302°F (150°C) for the minimum time necessary to ensure that the sample is completely fluid. Manually stir the sample but avoid incorporating air bubbles.

8.2 Pour 35 ± 0.5 g of the sample into each of the required glass containers, providing sufficient material for characterizing tests which are to be run on the residue.

8.3 Immediately after pouring the sample into a glass container, turn the container to a horizontal position. Rotate the container slowly for at least one full rotation, and attempt to pre-coat its cylindrical surface. It is not necessary to pre-coat the open end of the container, and care should be taken to prevent the sample from flowing out of the container during this step. Place the container horizontally in a clean cooling rack that is maintained in a draft-free, room-temperature location away from ovens and other sources of heat.

NOTE 3—Complete pre-coating may not be possible for certain binders.

NOTE 4—For maximum precision in determining mass change, the cooling rack should be in a location that is the same temperature and humidity as the balance used for measuring the mass of the containers.

NOTE 5—Static electricity may cause unstable mass measurements, due in part to the characteristics of the glass sample containers. This problem can be minimized by mounting a passive ion source inside the balance draft shield.[3]

[3] One such ion source is available as model 2U500 from NRD Inc., 2937 Alt Boulevard North, Grand Island NY 14072–1292.

TABLE 1 Precision of Test on Residue

Test Methods	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:				
Viscosity at 140°F (60°C)	...	...	2.3	6.5
Ductility at 60°F (15.6°C)[A]	3 cm	9 cm	...	...
Multilaboratory precision:				
Viscosity at 140°F (60°C)	...	...	4.2	11.9
Ductility at 60°F (15.6°C)[A]	6 cm	16 cm	...	...

[A] This is based on the analysis of data resulting from tests by 16 laboratories on two asphalts ranging from 13 to 30 cm.

8.3.1 Allow the glass sample containers to cool in the cooling rack for a minimum of 60 min, and a maximum of 180 min.

8.3.2 When mass change is being determined, use two separate containers for this determination. After cooling, determine the mass of these containers using an analytical balance having a resolution of 0.001 g or better. Separately place each container vertically on the balance, and record the mass to the full resolution of the balance.

8.4 With the oven at operating temperature and the airflow set at 4000 ± 200 mL/min, arrange the containers holding the asphalt in the carriage so that the carriage is balanced. Fill any unused spaces in the carriage with empty containers. Close the door and rotate the carriage assembly at a rate of 15 ± 0.2 r/min. Maintain the samples in the oven with the air flowing and the carriage rotating for 85 min. The test temperature of 325 ± 1°F (163 ± 0.5°C) shall be reached within the first 10 min; otherwise, discontinue the test.

8.5 At the conclusion of the testing period, remove any samples for mass change determination and place them horizontally in the cooling rack. Then, remove each remaining glass sample container, one at a time, and transfer its contents to a collection container having a capacity at least 30 % greater than the total expected volume of residue. This transfer shall be accomplished by first pouring out any residue that will flow freely from the glass sample container and then scraping out as much of the remaining residue as practical. While the residue is being removed from each sample container, the oven door shall remain closed, with the heater power on, the air on, and the remaining samples rotating in the carriage. The final container shall be removed from the oven within 5 min of removal of the initial container.

NOTE 6—Any scraping tool or technique may be used, as long as an average of 90 % or more of the residue is removed from the sample containers. It has been determined that circumferential scraping tends to be more effective than lengthwise scraping.

8.6 After removing the residue from each of the glass containers, gently stir the collection container to homogenize the residue without introducing air into it. Test the residue within 72 h of performing the RTFO test.

8.7 If the mass change is being determined, allow the designated residue sample containers to cool on the cooling rack for a minimum of 60 min and a maximum of 180 min. After cooling, determine the mass of these container using an analytical balance having a resolution of 0.001 g or better. Separately place each container vertically on the balance, and record the mass to the full resolution of the balance. Note whether any sample appears to have flowed out of the bottle.

NOTE 7—Some labs have reported problems with the sample flowing from the bottle during the test. If this occurs, both oven level and bottle dimensions should be checked. Bottles with a small annular ring appear to be particularly susceptible to this problem. Bottles that do not comply with the dimensional requirements should be removed from service.

NOTE 8—To improve mass change precision, the containers used for determining mass change should be handled with clean gloves or tongs, and transfer to the balance should be done with tongs, to prevent contamination and temperature changes which could distort the mass measurement.

9. Report

9.1 Report the results from the RTFO test in terms of the physical changes in the asphalt brought about by this method. These values are obtained by performing appropriate ASTM tests on the asphalt before and after the RFTO test.

9.2 When determined, report the average mass change of the material in the two containers as a mass percent of the original material. Report this calculated result to the nearest 0.001 %. 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 usually will exhibit a mass gain, while samples with a high percentage of volatile components usually will exhibit a mass loss.

10. Precision and Bias

10.1 Criteria for judging the acceptability of the viscosity at 140°F (60°C) and the ductility at 60°F (15.6°C) test results on the residue after heating are given in Table 1. The values 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 values given in Column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests. The values 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 values 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.

10.2 The precision of mass change measurements has been estimated based on an analysis of AMRL data representing approximately 5900 repetitions of this test. the analysis indicates that the standard deviation of the test (1S) can be expressed as a function of the mass change (X) by using the following equations:

Mass Change (X)	Single Operator Standard Deviation (1S)	Multi-Lab Standard Deviation (1S)
If X ≤ -0.1%	1S = 0.013 – 0.070 (X)	1S = 0.020 – 0.200 (X)
If X > -0.1%	1S = 0.020	1S = 0.040

10.3 The 95 % confidence limit for the acceptable range of two results (D2S) can be determined by multiplying the standard deviation (1S) estimates given in 10.2 by a factor of 2.83.

10.4 This test method has no bias because the values determined are defined only in terms of the test method.

11. Keywords

11.1 aging; asphalt cement; rolling thin-film oven test (RTFOT)