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Standard Test Method for Asphalt Content of Hot-Mix Asphalt by Ignition Method[1]

This standard is issued under the fixed designation D 6307; 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 asphalt content of hot-mix asphalt (HMA) paving mixtures and pavement samples by removing the asphalt cement at 540°C by ignition in a furnace.

NOTE 1—Aggregate obtained by this test method may be used for sieve analysis. Particle size degradation may occur with some aggregates.

1.2 *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 limitation prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[2]

C 702 Practice for Reducing Samples of Aggregate to Testing Size[2]

D 75 Practice for Sampling Aggregates[3]

D 140 Practice for Sampling Bituminous Materials[3]

D 979 Practice for Sampling Bituminous Paving Mixtures[3]

D 1461 Practice for Moisture or Volatile Distillates in Bituminous Paving Mixtures[3]

D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Testing Soil, Rock, and Related Construction Materials[4]

3. Summary of Test Methods

3.1 The asphalt cement in the paving mixture is ignited using the furnace equipment applicable to the particular method. The asphalt content is calculated by difference from the mass of the residual aggregate and moisture content. The asphalt content is expressed as mass percent of moisture-free mixtures. Test Method A is intended for furnaces with an internal, automated weighing system. Test Method B is intended for furnaces without an internal weighing system.

4. Significance and Use

4.1 This test method can be used for quantitative determination of asphalt content in hot-mix asphalt (HMA) paving mixtures and pavement samples for quality control, specification acceptance, and mixture evaluation studies. This test method does not require the use of solvents.

5. Apparatus

5.1 *Balance*, readable to 0.1 g, and capable of measuring the mass of sample, sample trays, and catch pan. The balance shall conform to the requirement of Specification D 4753, Class GP2.

5.2 *Sample Tray(s)*, of appropriate size that allows the samples to be spread thinly and allows air to flow up through and around the sample particles. The sample shall be enclosed completely with screen mesh or perforated stainless steel plate or other suitable material.

NOTE 2—Screen mesh or other suitable material with maximum and minimum openings of 3.35 mm and 600 microns, respectively, has been found to perform well.

5.3 *Catch Pan*, of appropriate size to hold the sample trays so that aggregate particles and melting asphalt binder falling through the screen mesh are caught.

5.4 *Catch Pan/Sample Tray(s) Handling Apparatus*, suitable for inserting catch pan and sample tray(s) into furnace and removing hot catch pan and sample tray(s) from furnace.

5.5 *Assorted Spatulas, Pans, Bowls, and Wire Brushes*, for preparing hot-mix asphalt mixtures and removing aggregate from sample tray(s) and catch pan.

5.6 *Protective Gloves*, well insulated and capable of withstanding 580°C.

5.7 *Ovens*—Mechanical ovens, convection or forced draft, shall be provided for drying aggregates and HMA mixtures, and preheating HMA mixtures prior to ignition testing.

5.8 *Ignition Furnace*, as described in 8.1.1 or 11.1.1.

6. Hazards

6.1 The temperature of the furnace, sample, sample tray(s), and catch pan after removal from the furnace is extremely high. Caution, therefore, must be exercised at all times when handling these items as failure to do so could result in serious injury, severe burns or fire. The sample, sample tray(s), and catch pan should be placed inside a safety cage and should not be allowed to cool near any materials which are subject to

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

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

[4] *Annual Book of ASTM Standards*, Vol 04.08.

ignition at the high temperatures used in this procedure. Furnace manufacturer's instruction manual must be followed to take all necessary precautions.

7. Sampling

7.1 Obtain samples of aggregate in accordance with Practice D 75.

7.2 Obtain samples of hot-mix asphalt in accordance with Practice D 979.

7.3 *Preparation of Test Specimens*:

7.3.1 If the mixture is not soft enough to separate with a spatula or trowel, place it in a large, flat pan and warm in an oven set at 110±5°C until it can be separated or mixed. Split or quarter the material in accordance with Practice C 702 until the mass of material required for the test is obtained.

7.3.2 The size of the test sample shall be governed by the nominal maximum aggregate size of the mixture and shall conform to the mass requirement shown in Table 1 (see Note 3).

NOTE 3—When the mass of the test specimen exceeds the capacity of the equipment used (for a particular method), the test specimen may be divided into suitable increments, tested, and the results combined for calculation of asphalt content based on the weighted average of the masses used in the increments.

7.4 Obtain samples of asphalt cement in accordance with Practice D 140.

TEST METHOD A

8. Apparatus

8.1 In addition to the apparatus listed in Section 5, the following apparatus is required for Test Method A.

8.1.1 *Furnace*, having a minimum temperature capability of 580°C and having an internal weighing system capable of measuring the mass of sample sizes of at least 2500 g. The furnace chamber shall be of sufficient size to accommodate sample sizes of at least 2500 g. A data collection system also shall be included so that the sample mass loss can be determined automatically to an accuracy of 0.1 g and displayed during a test. The test is deemed complete when the difference between consecutive measured mass loss does not exceed 0.01 % of the sample mass for three consecutive 1-min intervals. The equipment shall provide a printout of the test results. A system capable of reducing furnace emissions to an acceptable level also shall be incorporated in the furnace. The furnace shall be vented into a hood or to the outside and when set-up properly will have no noticeable odors escaping into the laboratory. Furnace will have fan with capability to pull air through the furnace to expedite the test and to reduce escape of smoke into laboratory. The furnace shall be equipped so that the door cannot be opened during the ignition test.

8.1.2 *Filters*, if required, of the type specified by the furnace manufacturer.

TABLE 1 Size of Sample

Nominal Maximum Aggregate Size Standard, mm	Minimum Mass of Sample, kg
4.75	0.5
9.5	1
12.5	1.5
19.0	2
25.0	3
37.5	4

9. Calibration

9.1 The results of this test method may be affected by the type of aggregate in the mixture because different aggregates lose mass on ignition to varying degrees. The results also may be affected by the presence of additives and modifiers in the HMA sample. Accordingly, to optimize accuracy, a calibration factor shall be established by testing three calibration samples for each mix type. The calibration shall be performed on a prepared sample of asphalt mixture, which also shall include additives and modifiers, if any, to be used.

9.2 Obtain samples of blended aggregate to be used in HMA in accordance with 7.1. The sample should be approximately the same mass and gradation as that to be used for the HMA test sample (10.1).

9.3 Obtain samples of asphalt cement to be used in HMA in accordance with 7.4.

9.4 Oven-dry the aggregate samples to a constant mass.

9.5 Set the furnace temperature to 540±5°C for calibration using mixtures.

9.6 Heat the aggregates and asphalt cement to approximately 150°C. Heat all mixing bowls and tools to approximately 150°C.

9.7 Prior to mixing of calibration samples, an initial or "butter" mix is required to condition the mixing equipment. Remove and discard the "butter" mix from the bowl by scraping, leaving a uniform coating of asphalt mix residue.

NOTE 4—The "butter" mix prevents calibration samples from being biased by residual asphalt mix retained in the mixing bowl.

9.8 Prepare three calibration samples at the design asphalt cement content (P). Incorporate additives and modifiers, if any, to be used.

9.9 Determine and record the mass of the sample tray(s) and catch pan to the nearest 0.1 g.

9.10 Evenly distribute the sample in the sample tray(s).

9.11 Determine the mass of the sample, sample tray(s), and catch pan to the nearest 0.1 g. Calculate and record the initial mass of the sample (M_I).

9.12 Heat the calibration sample in the furnace at 540±5°C until the change in mass of the sample during three consecutive 1-min intervals does not exceed 0.01 % of the sample mass (M_I).

9.13 Measure and record the mass (M_L) of the sample after ignition to the nearest 0.1 g. The mass can be obtained immediately upon completion of the test from printout or display.

9.14 Calculate the calibration factor (C_F) as follows:

$$C_F = \left(\frac{M_I - M_L}{M_I} \times 100\right) - P \quad (1)$$

where:

M_I = total mass of the mixture calibration sample prior to ignition,

M_L = total mass of the mixture calibration sample after ignition, and
P = percentage of actual asphalt cement in the mix by mass of the total mix expressed as a percentage.

9.15 Repeat these steps for two additional calibration samples. Calculate the average calibration factor (C_F) by averaging the three C_F values.

9.16 The temperature for testing HMA samples in 10.3 shall be the same temperature selected for testing mixture calibration samples.

10. Procedure

10.1 Obtain an HMA sample in accordance with the Section 7. The sample mass should be approximately the same as that used for calibration (9.2).

10.2 Oven-dry the HMA sample to constant mass at a temperature of 110±5°C or determine the moisture content of samples according to Practice D 1461 so that the measured mass loss can be corrected for moisture.

10.3 Set the furnace temperature to 540±5°C. Samples can be placed in the furnace at significantly lower temperatures since the furnace will quickly heat to the desired temperature once the sample begins to burn. The furnace temperature is likely to increase during the ignition phase of the test.

10.4 Determine and record the mass of the sample tray(s) and catch pan to the nearest 0.1 g.

10.5 Evenly distribute the sample in the sample tray(s).

10.6 Determine the mass of the sample, sample tray(s), and catch pan to the nearest 0.1 g. Calculate and record the initial mass of the sample (M_B).

10.7 Heat the sample in the furnace at the specified temperature until the difference between consecutive measured mass loss does not exceed 0.01 % of the sample mass (M_B) for three consecutive 1-min intervals. This point shall be determined automatically by the furnace's data collection system.

10.8 The furnace's data collection system shall measure and record automatically the aggregate mass (M_A) of the sample after ignition to the nearest 0.1 g. The mass shall be obtained immediately upon completion of tests by subtracting the mass loss measured by the furnace from the initial mass of the mix (M_B).

10.9 The corrected asphalt content shall be calculated automatically by the furnace's data collection system as follows:

$$\% AC = \left(\frac{M_B - M_A}{M_B} \times 100\right) - C_F \quad (2)$$

where:
AC = measured asphalt content percent by mass of the oven-dry HMA sample,
M_A = total mass of aggregate remaining after ignition,
M_B = total mass of the HMA sample prior to ignition, and
C_F = calibration factor obtained in Section 9 and entered into the furnace's data collection system.

TEST METHOD B

11. Apparatus

11.1 In addition to the apparatus listed in Section 5, the following apparatus is required for Test Method B.

11.1.1 *Furnace*, having a minimum temperature capability of 580°C and equipped with a fan capable of pulling air through the furnace to expedite the test and to reduce escape of smoke into the laboratory. The furnace chamber shall be of sufficient size to accommodate samples sizes of at least 2500 g. A system capable of reducing furnace emissions to an acceptable level also shall be incorporated in the furnace. The furnace shall be vented into a hood or to the outside, and when set up properly, will have no noticeable odors escaping into the laboratory. The furnace shall be equipped so that the door cannot be opened during the ignition test.

11.1.2 *Filters*, if required, of the type specified by the furnace manufacturer.

12. Calibration

12.1 The results of this test method may be affected by the type of aggregate in the mixture because different aggregates lose mass on ignition to varying degrees. The results also may be affected by the presence of additives and modifiers in the HMA sample. Accordingly, to optimize accuracy, a calibration factor shall be established by testing three calibration samples for each mix type. The calibration shall be performed on a sample of prepared asphalt mixture, which also shall include additives and modifiers, if any, to be used.

12.2 Obtain samples of blended aggregate to be used in HMA in accordance with 7.1. The sample should be approximately the same mass and gradation as that to be used for the HMA test sample (13.1).

12.3 Obtain samples of asphalt cement to be used in HMA in accordance with 7.4.

12.4 Oven-dry the aggregate samples to a constant mass.

12.5 Set the furnace temperature to 540±5°C for calibration using mixtures.

12.6 Heat the aggregates and asphalt cement to approximately 150°C. Heat all mixing bowls and tools to approximately 150°C.

12.7 Prior to mixing of calibration samples, an initial or "butter" mix is required to condition the mixing equipment. Remove and discard the "butter" mix from the bowl by scraping, leaving a uniform coating of asphalt mix residue.

NOTE 5—The "butter" mix prevents calibration samples from being biased by residual asphalt mix retained in the mixing bowl.

12.8 Prepare three calibration samples at the design asphalt cement content. Incorporate additives and modifiers, if any, to be used.

12.9 Determine and record the mass of the sample tray(s) and catch pan to the nearest 0.1 g.

12.10 Evenly distribute the sample in the sample tray(s).

12.11 Determine the mass of the sample, sample tray(s), and catch pan to the nearest 0.1 g. Calculate and record the initial mass of the sample (M_I).

12.12 Heat the calibration sample in the furnace at 540±5°C for at least 45 min.

12.13 Remove the sample from the furnace and allow it to cool for at least 10 min.

12.14 Measure and record the mass M_L of the sample after ignition to the nearest 0.1 g.

12.15 Place the sample back into the furnace.

12.16 After the furnace reaches the set point temperature, heat the calibration sample for 15 min.

12.17 Remove the sample from the furnace and allow it to cool for at least 10 min.

12.18 Measure and record the mass (M_L) of the sample after ignition to the nearest 0.1 g.

12.19 Repeat the steps 12.15-12.18 until the change in measured mass (M_L) of the sample after ignition does not exceed 0.01 % of the initial sample mass (M_I).

12.20 Record the last value obtained for (M_L) as the mass (M_L) of the sample after ignition.

12.21 Calculate the calibration factor (C_F) as follows:

$$C_F = \left(\frac{M_I - M_L}{M_I} \times 100\right) - P \quad (3)$$

where:

M_L = total mass of the calibration sample after ignition,
M_I = total mass of the calibration sample prior to ignition, and
P = percentage of asphalt cement in the mix by mass of the total mix expressed as a percentage.

12.22 Repeat these steps for two additional calibration samples. Calculate the average calibration factor (C_F) by averaging the C_F values.

12.23 The temperature for testing HMA samples in 13.3 shall be the same temperature selected for testing mixture calibration samples.

13. Procedure

13.1 Obtain an HMA sample in accordance with Section 7. The sample mass should be approximately the same as that used for calibration (12.2).

13.2 Oven-dry the HMA sample to constant mass at a temperature of 110±5°C or determine the moisture content of samples according to Practice D 1461 so that the measured mass loss can be corrected for moisture.

13.3 Set the furnace temperature to 540±5°C. Samples can be placed in the furnace at significantly lower temperatures since the furnace will heat quickly to the desired temperature once the sample begins to burn. The furnace temperature is likely to increase during the ignition phase of the test.

13.4 Determine and record the mass of the sample tray(s) and catch pan to the nearest 0.1 g.

13.5 Evenly distribute the sample in the sample tray(s).

13.6 Determine the mass of the sample, sample tray(s), and catch pan to the nearest 0.1 g. Calculate and record the initial mass of the sample (M_B).

13.7 Heat the HMA sample in the furnace at 540±5°C for at least 45 min.

13.8 Remove the sample from the furnace after ignition and allow it to cool for at least 10 min.

13.9 Measure and record the mass (M_A) of the sample after ignition to the nearest 0.1 g.

13.10 Place the sample back into the furnace.

13.11 After the furnace reaches the set point temperature, heat the sample for at least 15 min.

13.12 Remove the sample from the furnace and allow it to cool for at least 10 min.

13.13 Measure and record the mass (M_A) of the sample after ignition to the nearest 0.1 g.

13.14 Repeat steps 13.10-13.13 until the change in measured mass (M_A) of the sample after ignition does not exceed 0.01 % of the initial sample mass (M_B).

13.15 Record the last value obtained for (M_A) as the mass (M_A) of the sample after ignition.

NOTE 6—Steps 13.10-13.15 may not be necessary if it can be demonstrated from the mix calibration data that constant mass could be achieved by heating the sample only once in the furnace. The type and mass of the HMA sample being tested should be reasonably close to those of the calibration sample.

13.16 Calculate the corrected asphalt content as follows:

$$\%\,AC\left(\frac{M_B - M_A}{M_B} \times 100\right) - C_F \quad (4)$$

where:

AC = measured asphalt content percent by mass of the oven-dry HMA sample,
M_A = total mass of aggregate remaining after ignition,
M_B = total mass of the HMA sample prior to ignition, and
C_F = calibration factor, obtained in Section 12.

14. Report

14.1 The report shall include the following information.

14.1.1 Date.

14.1.2 Identification of aggregate and mix type.

14.1.3 Test number.

14.1.4 Calibration data.

14.1.5 Mass of HMA sample before and after ignition (nearest 0.1 g).

14.1.6 Measured asphalt content (nearest 0.01 %).

14.1.7 Aggregate gradation, if performed.

15. Precision and Bias

15.1 The single-laboratory standard deviation for asphalt content has been found to be 0.04 %; therefore, results of two properly conducted tests by the same operator on the same aliquot samples of HMA should not differ by more than 0.11 % (Notes 7-9).

15.2 The multi-laboratory standard deviation for asphalt content has been found to be 0.06 %; therefore, the results of two properly conducted tests from two different laboratories on HMA samples from the same batch should not differ by more than 0.17 % (Notes 7-9).

15.3 The bias for this test method is currently under development.

NOTE 7—These numbers represent, respectively, the (1S) and D2S) limits described in Practice C 670.

NOTE 8—These precision statements are based on four aggregate types, four replicates, and 12 laboratories participating with no laboratory results deleted as outlying observations. All four aggregates had relatively low absorption values (water absorption values ranging from 0.5 to 0.8 %).

NOTE 9—These precision statements are applicable to Test Method A only, using laboratory prepared mixtures (not plant-mixed pavement samples). The precision of Test Method B has not been determined.

16. Keywords

16.1 asphalt content; asphalt paving mixtures; hot-mix asphalt; ignition method