Asphalt Content of Asphalt Mixture by Ignition Method

ASTM D6307 – 19

4. Significance and Use

4.1 This test method can be used for quantitative determination of asphalt content in asphalt mixture and pavement samples for quality control, specification acceptance, and mixture evaluation studies. This test method does not require the use of solvents. Aggregate obtained by this test method may be used for gradation analysis according to Test Method D5444.

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 be in accordance with Guide D4753, 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, 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 µm, 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 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 asphalt mixtures, and for preheating asphalt 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 that are subject to ignition at the high temperatures used in this procedure.....

The 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 D75/D75M.....

7.2 Obtain samples of asphalt mixture in accordance with Practice D979/D979M and AASHTO R47

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 AASHTO R47 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.

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

7.4 Obtain samples of asphalt cement in accordance with Practice D140/D140M.....

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 *Ignition Furnace*—A forced-air ignition furnace that heats the sample by either convection method or direct irradiation method.....

The convection-type furnace must have a minimum temperature capability of 580 °C.....

The furnace shall have 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.....

The furnace will have a fan with the capability to pull air through the furnace to expedite the test and to reduce escape of smoke into the 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.....

9. Calibration

9.1 The type of aggregate in the mixture may affect the results of this test method 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 asphalt mixture 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 the asphalt mixture in accordance with 7.1. The sample should be approximately the same mass and gradation as that to be used for the asphalt mixture test sample (10.1) .....

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

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

9.4.1 For the convection-type furnace, set the furnace temperature to 540 ± 5 °C for calibration using mixtures.....

9.4.2 For the direct irradiation-type furnace, set the burn profile to the DEFAULT mode.....

9.5 Heat the aggregates and asphalt cement to approximately 150 °C.....

Heat all mixing bowls and tools to approximately 150 °C.....

9.6 Prior to the 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.

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

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

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

9.10 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 (*MI*) .....

9.11 Heat the calibration sample in the convection-type furnace at 540 ± 5 °C; or in the direct irradiation-type furnace using the DEFAULT mode until the change in mass of the sample during three consecutive 1-min intervals does not exceed 0.01 % of the sample mass (*MI*) .....

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

9.13 Calculate the calibration factor (*CF*) as follows: .....

$$CF = \left(\frac{MI - ML}{MI} X\ 100\right) - P \qquad (1)$$

where:

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

ML = 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.

9.14 Repeat steps 9.8 – 9.13 for two additional calibration samples.....
Calculate the average calibration factor (*CF*) by averaging the three *CF* values.....
9.15 *Calibration Temperature Adjustments:*
9.15.1 For the convection-type furnace, if the calibration factor exceeds 1.0 %, lower the test temperature to 482 ± 5 °C and repeat steps 9.2 – 9.14.....
Use the calibration factor obtained at 482 °C, even if it exceeds 1.0 %.....
9.15.2 For the direct irradiation-type furnace, the DEFAULT burn profile should be used for most materials.....
The operator may select burn profile OPTION 1 or OPTION 2 to optimize the burn cycle.....
OPTION 1 is designed for aggregates with a correction factor greater than 1.0 %. OPTION 2 is designed for samples that may not burn completely using the DEFAULT burn profile.
9.16 The temperature or burn profile for testing asphalt mixture samples in 10.3 shall be the same temperature or burn profile selected for testing mixture calibration samples.....

10. Procedure

10.1 Obtain an asphalt mixture sample in accordance with Section 7. The sample mass should be approximately the same as that used for calibration (9.2) .....
10.2 Oven dry the asphalt mixture sample to constant mass at a temperature of 110 ± 5 °C or determine the moisture content of samples in accordance with Test Method D1461, so that the measured mass loss can be corrected for moisture.....
10.3 Set the furnace temperature or burn profile in accordance with Section 9.....
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 (*MB*) .....
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 (*MB*) 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 automatically record the aggregate mass (*MA*) 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 (*MB*) .....

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

$$\% AC = \left(\frac{MB - MA}{MB} X\ 100\right) - CF \quad (2)$$

where:
AC = measured asphalt content percent by mass of the oven-dry asphalt mixture sample,
MA = total mass of aggregate remaining after ignition,
MB = total mass of the asphalt mixture sample prior to ignition, and
CF = 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 may also be affected by the presence of additives and modifiers in the asphalt mixture 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 the asphalt mixture in accordance with 7.1. The sample should be approximately the same mass and gradation as that to be used for the asphalt mixture test sample (13.1) .....
12.3 Obtain samples of asphalt cement to be used in the asphalt mixture 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 the 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 (*MI*) .....
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 (*ML*) 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 (*ML*) of the sample after ignition to the nearest 0.1 g.....
12.19 Repeat 12.15 – 12.18 until the change in measured mass (*ML*) of the sample after ignition does not exceed 0.01 % of the initial sample mass (*MI*) .....
12.20 Record the last value obtained for (*ML*) as the mass (*ML*) of the sample after ignition.....
12.21 Calculate the calibration factor (*CF*) as follows: .....

$$CF = \left(\frac{MI - ML}{MI} X\ 100\right) - P \qquad (3)$$

where:
ML = total mass of the calibration sample after ignition,
MI = 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 (*CF*) by averaging the *CF* values.....
12.23 The temperature for testing the asphalt mixture samples in 13.3 shall be the same temperature selected for testing mixture calibration samples.....

13. Procedure

13.1 Obtain an asphalt mixture 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 asphalt mixture sample to constant mass at a temperature of 110 ± 5 °C or determine the moisture content of samples according to Test Method D1461, 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 (*MB*) ....._____

13.7 Heat the asphalt mixture 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 (*MA*) 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 (*MA*) of the sample after ignition to the nearest 0.1 g....._____

13.14 Repeat 13.10 – 13.13 until the change in measured mass (*MA*) of the sample after ignition does not exceed 0.01 % of the initial sample mass (*MB*) ....._____

13.15 Record the last value obtained for (*MA*) as the mass (*MA*) 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 asphalt mixture sample being tested should be reasonably close to those of the calibration sample.

13.16 Calculate the corrected asphalt content as follows: ....._____

$$\% AC = \left(\frac{MB - MA}{MB} X\ 100\right) - CF \qquad (4)$$

where:

AC = measured asphalt content percent by mass of the oven-dry asphalt mixture sample,

MA = total mass of aggregate remaining after ignition,

MB = total mass of the asphalt mixture sample prior to ignition, and

CF = calibration factor, obtained in Section 12.

14. Report

14.1 Report 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 the asphalt mixture 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....._____

Comments