Florida Method of Test for Quantitative Determination of Asphalt Content from Asphalt Paving Mixtures by the Ignition Oven

Designation: FM 5-563

1. SCOPE

This method covers the quantitative determination of asphalt content in asphalt concrete pavement mixtures and pavement samples by removal of the asphalt through ignition in a furnace at 538°C (1000°F). Aggregate remaining from this method may be used for sieve analysis using FM 1-T 030.

The values stated in metric (SI) units are to be regarded as standard. Values given in parenthesis are for information and reference purposes only.

This standard does not purport to address all the safety or environmental problems 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.

2. APPLICABLE DOCUMENTS

2.1 AASHTO Standards:


2.2 Florida Test Methods:

FM 1-T 030 Mechanical Analysis of Extracted Aggregate

FM 1-T 168 Sampling Bituminous Paving Materials

2.3 Furnace Manufacturer's User's Manual

3. SUMMARY OF METHOD

3.1 The asphalt content of a paving mixture is determined by burning off the asphalt by ignition at 538°C (1000°F). The asphalt content is calculated from the initial weight of the sample, the final weight of the remaining aggregate, a temperature compensation factor for the measurement system, and a calibration factor for the mix. The asphalt content is expressed as both a weight loss in grams, and as a weight percentage of the initial sample weight.
3.2 This test procedure contains provisions for the user to establish a mix calibration factor to account for the aggregate weight loss due to ignition for the particular mixture.

4. **APPARATUS**

4.1 Ignition Furnace - A forced-air furnace capable of maintaining a temperature setting of up to 650°C (1200°F). The furnace is to include an internally mounted balance or load cell, accurate to 0.1 g, which is thermally isolated from the furnace chamber. This balance shall be capable of weighing samples of at least 2500 g in addition to the weight of the sample baskets. An automated system shall also be included for data collection and processing. The system shall provide a printout of the initial specimen weight, specimen weight loss, temperature compensation factor, mix calibration factor, calibrated asphalt content (%), test duration time, and furnace set-point temperature. An exhaust with filter, afterburner, or some combination that reduces the furnace emissions to an acceptable level shall be incorporated into the furnace. The furnace shall also include an audible alarm and indicator light to signal the end of the test. The test is deemed complete when the measured weight loss does not exceed 0.01% of the total sample weight for three consecutive minutes. The furnace door must lock automatically when the test is initialized and remain locked until completion of the test.

**Note 1:** See precautions in Section 5.

4.2 Baskets - A minimum of two tempered stainless-steel baskets of perforated sheet or mesh. The mesh or perforations should be small enough to minimize the loss of sample during burn off. The baskets shall nest together and provide shielding on the corners to minimize loss of aggregate. The nested baskets shall sit in a catch pan approximately 25 mm (1 in.) deep. The bottom basket shall sit higher than 40 mm (1.6 in.) above the catch pan. The overall dimensions of the nested baskets in the catch pan shall be such that the assembly can be easily placed onto the balance tray inside the furnace without scraping the chamber sides. The pan shall incorporate a system (handle or tongs) to lift the basket assembly for placement into and removal from the furnace. This system shall minimize the exposure of the operator to the high temperature in the furnace.

4.3 Balance - A balance, with a capability of 8 kg or greater, accurate to 0.1 g. It shall conform to the requirements of AASHTO M231, Class G2.

4.4 Oven - An oven capable of maintaining a temperature of 171°C ± 5°C (340°F ± 9°F).
4.5 Safety Equipment - Safety face shield, high temperature gloves, and a long sleeve jacket. A heat resistant surface capable of withstanding temperatures up to 650°C (1200°F) and a protective cage to surround and isolate the sample baskets shall also be provided.

4.6 Miscellaneous Equipment - A pan with internal dimensions larger than the outside dimensions of the baskets, catch pan for transferring samples after ignition, wire and soft bristle brushes, spatulas, and bowls may be needed.

5. PRECAUTIONS

5.1 The oven must be placed in a well-ventilated area and properly vented to the outside (see manufacturer’s recommendations).

5.2 Prior to initiating the test, verify that the ignition oven door has been properly closed. This ensures that the door lock will engage when the test is started.

6. SAMPLING

6.1 The test samples shall be obtained in accordance with FM 1-T 168.

6.2 Preparation of test specimens:

   6.2.1 If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in a large, flat pan and heat to a temperature not to exceed 171°C (340°F) until it can be handled or mixed.

   6.2.2 The size of the test samples shall be governed by the nominal maximum aggregate size of the mixture and shall be as shown in Table 1.

   Note 2: When the sample size exceeds the capacity of the equipment used, the sample may be divided into suitable increments. The specimens may then be tested, and the results mathematically combined for calculation of the asphalt content (weighted average).
### Table 1

<table>
<thead>
<tr>
<th>Mixture Type</th>
<th>Minimum Sample Size (grams)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ATPB (No. 57 stone)</td>
<td>3000</td>
</tr>
<tr>
<td>SP-19.0</td>
<td></td>
</tr>
<tr>
<td>ABC-3</td>
<td></td>
</tr>
<tr>
<td>S-II</td>
<td></td>
</tr>
<tr>
<td>ATPB (No. 67 stone)</td>
<td>2000</td>
</tr>
<tr>
<td>SP-12.5</td>
<td></td>
</tr>
<tr>
<td>FC-12.5 (FC-6)</td>
<td></td>
</tr>
<tr>
<td>SP-9.5</td>
<td></td>
</tr>
<tr>
<td>FC-9.5</td>
<td></td>
</tr>
<tr>
<td>FC-5</td>
<td></td>
</tr>
<tr>
<td>S-I</td>
<td></td>
</tr>
<tr>
<td>FC-2 and FC-3</td>
<td></td>
</tr>
<tr>
<td>S-III</td>
<td></td>
</tr>
<tr>
<td>SP-4.75</td>
<td></td>
</tr>
<tr>
<td>FC-4.75</td>
<td></td>
</tr>
<tr>
<td>TYPE III</td>
<td></td>
</tr>
<tr>
<td>TYPE II</td>
<td></td>
</tr>
<tr>
<td>ABC-1 and ABC-2</td>
<td></td>
</tr>
<tr>
<td>SAHM</td>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

7. **MIXTURE CALIBRATION**

7.1 This test method may be affected by the type of aggregate used in the mixture. Accordingly, to optimize accuracy, an asphalt binder calibration factor must be established for each mix.

7.2 Two calibration specimens conforming to the size requirements of Section 6.2.2 shall be prepared at the design asphalt content. A “butter mix” shall also be prepared in each mixing bowl at the design asphalt content, mixed and discarded prior to mixing any of the calibration specimens to ensure an accurate asphalt content.

**Note 3:** The freshly mixed specimens may be placed directly in the sample baskets. If allowed to cool, the samples must be pre-heated in an 171°C (340°F) oven until workable. Do not preheat the sample baskets.

7.3 Weigh and record the weight of the sample baskets and catch pan (with guards in place).

7.4 Place the bottom sample basket in the catch pan. Evenly distribute approximately one half of the calibration specimen in the lower basket, keeping the material approximately 25 mm (1 in) away from the edges of the basket.
7.5 Place the upper sample basket on the bottom basket assembly. Evenly distribute the remaining specimen in the top basket as in Section 7.4.

**Note 4:** If the basket assembly contains more than two baskets, divide the sample into equal proportions and distribute them equally in each one of the sample baskets.

7.6 Weigh the sample and the sample basket assembly. Subtract from this weight the weight measured in Section 7.5 and record. This is the initial weight, $W_i$, of the sample specimen.

7.7 Preheat the ignition furnace to 538°C (1000°F).

7.8 Enter a calibration factor of 0.00 in the ignition furnace.

7.9 Input the initial weight, $W_i$ of the sample specimen, rounded to the nearest gram, into the ignition furnace controller. Verify that the correct weight has been entered.

7.10 Using protective equipment, open the chamber door and place the sample basket assembly in the furnace. Ensure that the basket assembly is not touching the furnace sides. Close the chamber door and initiate the test by pressing the start/stop button.

7.11 Allow the test to continue until the stable light and audible stable alarm indicate that the test is complete. Press the start/stop button. Print out the test results.

7.12 Using protective equipment, open the chamber door, remove the sample baskets, place on an insulated heat resistant surface and cover the basket assembly with the protective cage. Allow to cool to room temperature (approximately 30 minutes), and then determine the final weight, $W_f$, of the sample specimen, as in Section 7.6, before discarding or performing a gradation analysis, if required.

7.13 Once the testing of a calibration specimen has been completed, the actual percent weight loss attributed to the aggregate in the mix shall be determined as follows:

$$W_L = AC_{Actual} - AC_{Measured}$$

where:

- $W_L =$ aggregate weight loss of the calibration sample, percent by weight of the total mix;
- $AC_{Actual} =$ percent asphalt in the mix, by weight of the total mix, before ignition (design asphalt content as
per Section 7.2).

\[ \text{AC}_{\text{Measured}} = \text{percent asphalt in the mix, by weight of the total mix, after ignition as given in the furnace printout.} \]

7.14 The asphalt binder calibration factor is calculated as follows:

\[ CF[AC] = \frac{W_{L1} + W_{L2}}{2} \]

where:
- \( CF[AC] \) = asphalt binder calibration factor, percent by total weight of mix;
- \( W_{L1} \) = aggregate weight loss of the first calibration sample, percent by weight of the total mix;
- \( W_{L2} \) = aggregate weight loss of the second calibration sample, percent by weight of the total mix.

8. **TESTING PROCEDURE**

8.1 Preheat the ignition furnace to 538°C (1000°F).

8.2 Enter a correction factor of 0.00 in the ignition furnace.

8.3 Weigh and record the weight of the sample baskets and catch pan (with guards in place).

8.4 Prepare the sample as described in Section 6. Place the bottom sample basket in the catch pan. Evenly spread approximately one half of the test specimen in the lower basket, keeping the material approximately 25 mm (1 in) away from the edges of the basket.

8.5 Place the upper sample basket on the bottom basket assembly. Evenly distribute the remaining specimen in the top basket as in Section 8.4.

8.6 Weigh the sample and the sample basket assembly. Subtract from this weight the weight measured in Section 8.3 and record. This is initial weight, \( W_i \), of the sample specimen.

8.7 Input the initial weight, \( W_i \), of the sample specimen, rounded to the nearest gram, into the ignition furnace controller. Verify that the correct weight has been entered.

8.8 Using protective equipment, open the chamber door and place the sample basket assembly in the furnace. Ensure that the basket assembly is not touching the furnace sides or back of the furnace. Close the chamber.
door and initiate the test by pressing the start/stop button.

8.9 Allow the test to continue until the stable light and audible stable alarm indicates the test is complete. Press the start/stop button. This will initiate the printout of the test results and stop the test.

8.10 Using protective equipment, open the chamber door, remove the sample baskets, and place on an insulated heat resistant surface. Cover the basket assembly with the protective cage and allow to cool to room temperature (approximately 30 minutes). The final weight $W_F$, of the sample specimen may then be determined, as in Section 8.6.

8.11 If required or necessary, perform a gradation analysis on the residual aggregate as indicted in Section 10.

**Note 5:** If the basket assembly contains more than two baskets, divide the sample into equal proportions and distribute them equally in each one of the sample baskets.

9. **CALCULATIONS**

9.1 The calibrated asphalt content shall be computed as follows:

$$AC_{Calibrated} = AC_{Measured} + CF[AC]$$

where: $AC_{Calibrated}$ = calibrated asphalt content, percent by weight of the total mix;

$AC_{Measured}$ = measured asphalt content as obtained from the furnace printout, percent by weight of the total mix;

$CF[AC]$ = asphalt binder calibration factor of the specific mix being tested as determined in accordance with Section 7, percent by weight of the total mix.

9.2 If the furnace printout is not available due to an equipment issue, and if approved by the Engineer, the measured AC content may be calculated manually as follows:

$$AC_{Measured} = \frac{W_i - W_F}{W_i} \times 100$$

where: $W_i$ = initial weight of the sample

$W_F$ = final weight of the sample.
10. **GRADATION**

10.1 After allowing the sample to cool to room temperature, empty the contents of the baskets into a flat pan. Use a small wire sieve brush, and/or a bristle brush to ensure that any residual fines are removed from the baskets.

10.2 Perform a gradation analysis according to FM 1-T 030.

11. **REPORTING**

11.1 Report the calibrated asphalt content, mix calibration factor, temperature compensation factor, total percent loss, sample weight, and test temperature. Attach the original printed ticket to the report.

**Note 6:** The thermal paper used for some of these machines may darken beyond legibility when exposed to heat or after handling and aging. It is recommended that a copy be made of the ticket to prevent the loss of this information.

12. **PRECISION AND BIAS**

12.1 Precision (plant produced mix)

The following precision statements were determined in a round-robin study and were based on nine plant produced surface mixtures containing a wide variety of aggregate types. There were four replicates per mix type with twelve laboratories participating.

<table>
<thead>
<tr>
<th></th>
<th>Standard Deviation (1S)</th>
<th>Acceptable Range of Two Test Results (D2S)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>% Asphalt Content</td>
<td>% Asphalt Content</td>
</tr>
<tr>
<td>Single-Operator Precision</td>
<td>0.114</td>
<td>0.32</td>
</tr>
<tr>
<td>Multi-Laboratory Precision</td>
<td>0.156</td>
<td>0.44</td>
</tr>
</tbody>
</table>
12.2 Precision (laboratory fabricated samples)

The following precision statements were determined in a round-robin study and were based on six laboratory fabricated surface mixtures containing six aggregate types. There were three replicates per mix type with twelve laboratories participating. The results from one laboratory were deleted as outlying observations.

<table>
<thead>
<tr>
<th></th>
<th>Standard Deviation (1S) % Asphalt Content</th>
<th>Acceptable Range of Two Test Results (D2S) % Asphalt Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single-Operator Precision</td>
<td>0.047</td>
<td>0.13</td>
</tr>
<tr>
<td>Multi-Laboratory Precision</td>
<td>0.141</td>
<td>0.35</td>
</tr>
</tbody>
</table>

12.3 Bias

The bias of this test method has not been determined.