

**METHOD OF TEST FOR
QUANTITATIVE DETERMINATION OF ASPHALT CEMENT CONTENT
BY IGNITION AND ANALYSIS OF REMAINING AGGREGATE
FROM BITUMINOUS PAVING MIXTURES**

1. SCOPE

1.1 This method covers the quantitative determination of asphalt cement in a bituminous paving mixture by ignition in a furnace. It can be used for mixes containing recycled asphalt pavement (RAP). The method requires that the furnace be calibrated using materials that are blended proportionally in the laboratory to duplicate the paving mixture.

1.2 The method can also be used to determine the gradation of the aggregate remaining after the burn.

2. REFERENCES

2.1 MTO Test Methods

LS-282 Quantitative Extraction of Asphalt Cement and Analysis of Extracted Aggregate from Bituminous Mixtures, or LS-291 (same title, Ontario Method)

LS-312 Procedure for the Fractionation of Unextracted Recycled Asphalt Pavement (RAP) for Testing and for Incorporating in Other Test Samples

2.2 ASTM Standards

D6307-05 Standard Test Method for Asphalt Content of Hot-Mix Asphalt by Ignition Method

2.3 Furnace manufacturer's instruction manual

3. METHOD

3.1 ASTM D6307-05, Standard Test Method for Asphalt Content of Hot-Mix Asphalt by Ignition Method, shall be followed, except as amended below.

4. AMENDMENTMENTS TO ASTM D6307-05

4.1 APPARATUS

Section 8.1.1 of ASTM D6307 is replaced with the following:

FURNACE: Heats the sample by either the convection method or direct irradiation method. The convection type furnace shall have a minimum temperature capability of 580°C. The furnace chamber 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 shall also be included so that the sample mass loss can be automatically determined to an accuracy of 0.1 g and displayed during a test. The equipment shall

provide a printout of the test results. The equipment shall be capable of varying the test end point from 0.1 g to 1.0 g on a sample size of 1500 g (0.007% to 0.07% on 1500 g). After the testing end point criteria are met, the furnace shall be capable of alerting the operator and/or turning off the furnace heating elements. A system capable of reducing furnace emissions to a level meeting, or exceeding all Health and Safety and environmental standards, shall also be incorporated in the furnace. The furnace shall be vented into a hood or to the atmosphere and, when set up properly, will have no noticeable odours 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.

4.2 CALIBRATION

Section 9. Calibration of ASTM D6307 is modified as follows:

To Clause 9.2, add:

Each constituent aggregate shall be fractioned on all sieves down to the 2.36 mm sieve, with the passing 2.36 mm sieve collected as a single fraction. Prepare the calibration samples by reconstituting to the JMF gradation up to and including the 2.36 mm sieve using the proportions indicated in the mix design. The total fraction passing the 2.36 mm shall be obtained by drawing from the passing 2.36 mm of each constituent in the proportion of its source material as per the mix design. For recycled mixes, include the amount of RAP calculated on the basis of the mix design to the aggregate blend. The RAP shall be prepared in accordance with LS-312.

To Clause 9.7, add:

If RAP is a constituent of the mix, the design asphalt content includes AC from this RAP. Allowance shall be made for this quantity of asphalt cement already in the aggregate blend by determining the AC content of the RAP through solvent extraction (Methods LS-282 or LS-291). The gradation of the extracted RAP shall also be carried out as part of this calibration.

Certain aggregate types may result in an unusually high calibration factor and erroneous gradation results due to aggregate breakdown. It is, therefore, necessary to prepare the requisite calibration samples for AC content and two additional "blank" samples for gradation calibration (to which no asphalt cement will be added and which will not be subject to burning in the furnace). For recycled mixes, RAP shall not be incorporated in the blank (gradation calibration) samples.

Once the blank samples have been prepared, carry out a washed sieve analysis of each of the "blank" samples and obtain the mean gradation. For mixes containing no RAP, this will be the gradation to which the gradation of the burnt calibration samples will be compared to calculate the correction factors for each sieve. For recycled mixes, the mean gradation of the blank samples will be mathematically combined with the gradation of RAP (determined after solvent extraction).

Replace Clause 9.13 with the following: A "calibration factor_{lab mix}" for asphalt cement content is the difference between an ignition furnace test result produced using raw materials that are blended in the laboratory, in accordance with the job mix formula (JMF) requirements and the asphalt content per cent required by the JMF, where: $\text{calibration factor}_{\text{lab mix}} = \text{ignition result}_{\text{using JMF \%AC}} - \%AC_{\text{JMF}}$.

Clause 9.11 is replaced with the following: The furnace end point shall be set to 1.0 g for all sample sizes (0.2% by mass at 500 g sample size; 0.07% at 1500 g; 0.05% at 2000 g). The test shall be deemed to be complete when the difference between measured loss for 3 consecutive 1 min intervals does not exceed 1.0 g.

For a convection type furnace, the testing temperature chosen shall be the highest of 430°C, 480°C, or 540°C, providing that individual "calibration factors_{lab mix}" are less than 1.0%. For a direct irradiation type furnace, follow the operating instructions provided by the device manufacturer; including setup of the burn profile for the type of mix being tested, providing that individual "calibration factors_{lab mix}" are less than 1.0%. To optimize accuracy, a "mean calibration factor_{lab mix}" must be obtained for each mix type. The "mean calibration factor_{lab mix}" shall be the average of three "calibration factors_{lab mix}" where the range for the 3 tests is less than or equal to 0.15%. If the range is greater than 0.15%, another two tests shall be done and, from the 5 tests, the highest and lowest results shall be discarded and the remaining 3 "calibration factors_{lab mix}" averaged. The calibration factor is unique for a particular oven and the mix, and must be established before any quality control, acceptance, or referee testing is completed.

To determine correction factors for gradation, perform a gradation analysis on each of the 3 residual aggregate samples (in accordance with Section 4.7) and obtain the average gradation resulting from the ignition process. A "calibration factor_{lab mix}" for gradation for any sieve is the difference between the gradation of an ignition furnace test result produced using raw materials that are blended in the laboratory, in accordance with the job mix formula (JMF) requirements, and the gradation of the blank unburnt sample.

A gradation correction factor is only applied when it is greater than or equal to 1.0% for any sieve except the 75 µm sieve. For this sieve, the factor shall be applied if it is greater than or equal to 0.3%.

4.3 PREPARATION OF SAMPLE

Section 7. Sampling is replaced with the following:

7.1 Use a sample obtained according to approved methods. Large field samples shall be reduced to the appropriate size for testing. Two methods of reducing the field sample to the testing size are acceptable: quartering or splitting using a riffle splitter.

7.1.1 Quartering: Warm the field sample to achieve sufficient workability for quartering. A conventional oven maintained at 110°C or a microwave oven (see Note 1) may be used for this purpose. The surface upon which the sample is to be reduced should be flat, non-abrasive,

non-absorptive, and of sufficient area to provide for uniform quartering. Heat lamps may be used to keep the surface warm (see Note 2). The sample shall be mixed on the surface until uniform, then quartered, and opposite quarters removed. This process is to be repeated until the testing size is obtained.

Note 1: *Caution: Frequent mixing may be necessary to prevent localized overheating when using a microwave oven to heat paving mixtures. Also, the presence of metallic particles in some mixtures may render the microwave oven unsafe for the heating of these mixtures. Mixes made with polymer modified asphalt cement will have to be heated to a temperature higher than 110°C to achieve sufficient workability. The actual temperature should be the minimum required to achieve this and should be determined by trial and error.*

Note 2: *If required, 'Pam' or equivalent used in minimal quantities has been found suitable in preventing the sample from adhering to the surface. Motor oils must not be used for this purpose.*

7.1.2 Riffle Splitter: Warm the field sample to achieve sufficient workability for splitting. A conventional oven maintained at 110°C or a microwave oven (see Note 1 above) may be used for this purpose. The mix may agglomerate if it is too cold, or stick to the splitter if too hot. A temperature range of 90°C to 110°C has been found to be generally satisfactory. Heat lamps may be used to keep the walls of the splitter box warm (see Note 2 above). Chutes shall be cleaned after each split. The use of a putty knife or a 25 mm diameter wire brush used to clean glassware has been found suitable.

7.2 When testing core samples for asphalt cement content and sieve analysis, the core shall be warmed just sufficiently to enable the trimming of the curved surface to remove particles that were cut during the coring process. The depth trimmed shall be equivalent to the 'Designated Large Sieve' size for the particular mix type, except for HL 2 mix, for which the depth trimmed shall be approximately 4.75 mm. Dry the trimmed core to constant mass to remove moisture.

7.3 A single test portion shall be used to determine the asphalt cement content. The sample size for testing is governed by the 'Designated Large Sieve' size of the mix (see Note 3) and shall conform to the requirements as shown in Table 1.

Table 1 - Size of Test Sample

Designated Large Sieve Size, MTO Sieve Designation, mm	Minimum Mass of Sample, kg
2.36	0.5
9.5	1.5
13.2	1.5
12.5/16.0/19.0	2.0
25.0	3.0

Note 3: The Designated Large Sieve is a sieve size specifically designated for the following mixes for gradation testing:

2.36 mm for HL 2;

9.5 mm for HL 1, HL 3, HL 3A, Dense Friction Course or Open Friction Course, Superpave 12.5, 12.5FC 1 and 12.5FC 2;

12.5 mm for Superpave 19.0;

13.2 mm for HL 4 Binder or Surface Course;

16.0 mm for HL 8, Medium Duty Binder Course, or Heavy Duty Binder Course;

19.0 mm for Superpave 25.0; and

25.0 mm for Superpave 37.5.

The gradation analysis shall be performed on the total test portion (see Section 4.7).

7.4 When splitting off a sample for determination of moisture content, a minimum sample size of 1000 g is required for all types of paving mix.

7.5 Determination of Moisture Content

7.5.1 If moisture is present in a sample, it will appear as asphalt cement after ignition testing and internal furnace software calculations are completed, unless a correction is made. This is done by determining the amount of moisture in a 1000 g portion and:

- a) adjusting the asphalt cement content per cent after ignition testing or if the per cent moisture is known,
- b) adjusting the mean calibration factor before ignition testing.

The first approach has the advantage that the two procedures can be run concurrently. Either way is acceptable.

The weighing and recording of the masses for both the moisture content determination and the ignition tests must be done at the same time - immediately before testing for ignition. In either case, weigh the test portion to be used and record the mass to 0.1 g. Place the test portion for moisture determination in an oven maintained at $110 \pm 5^{\circ}\text{C}$ and dry to constant mass.

Note 4: Constant mass is defined as no change in mass in excess of 0.1% or 1 g, whichever is less, for each 30 min drying period.

7.5.2 Reweigh the test portion and calculate its moisture content rounding off to the nearest 0.01% as follows:

$$\% \text{ Moisture Content, (Z)} = \frac{(M_a - M_d)}{M_d} \times 100$$

Where: M_a = Original mass of mix, g

M_d = Mass of oven dry mix, g

4.4 PROCEDURE

Section 10. Procedure of ASTM D6307 is amended as follows:

Clause 10.3 is amended by the addition of the following: For the convection type furnace, testing temperatures shall be one of $430 \pm 5^{\circ}\text{C}$, $480 \pm 5^{\circ}\text{C}$, or $540 \pm 5^{\circ}\text{C}$, as determined by Furnace Calibration (Section 4.2). For the direct irradiation type furnace, the burn profile for testing HMA samples shall be the same burn profile selected for testing mixture calibration samples (Section 4.2). The following follow Clause 10.9 of ASTM D6307-05:

10.10 The convection furnace temperature or direct irradiation furnace burn profile used for quality control, acceptance, or referee testing must be the same as that at which the "mean calibration factor_{lab mix}" is determined.

10.11 After loading the test portion into the furnace and during test initiation procedures, input the testing mass into the furnace software. Also input the mean calibration factor into the furnace software as given below.

10.11 For dry field samples, input the "mean calibration factor_{lab mix}" into the furnace software.

If a) of Section 7.5.1 is used, input the "mean calibration factor_{lab mix}" into the furnace software.

If b) of Section 7.5.1 is used, input the "mean calibration factor_{field mix}" into the furnace software, where:

$$\text{Mean Calibration Factor}_{\text{field mix}} = \text{Mean Calibration Factor}_{\text{lab mix}} + \% \text{ Moisture Content}$$

Because of per cent moisture content, this "mean calibration factor_{field mix}" may be greater than 1.0%.

4.5 ASPHALT CEMENT CONTENT CALCULATION

Calculate the asphalt cement content in the test portion to 2 decimal places making use of worksheets.

4.5.1 If the ignition furnace test sample is dry, the ignition furnace result will be the % AC_{correct}.

4.5.2 If a) of Section 4.4.1 was used, adjust the asphalt cement content for the test portion as follows:

$$\% \text{ AC}_{\text{correct}} = \% \text{ AC}_{\text{by ignition}} - \% \text{ Moisture}$$

4.5.3 If b) of Section 4.4.1 was used, the ignition furnace result will be the % AC_{correct}.

4.6 ANALYSIS OF RESIDUAL AGGREGATE

4.6.1 Washing of Aggregate: After the mass (W_3) of the test portion of dried aggregate has been determined, return the sample to the drying pan and cover the sample with water. Add a sufficient amount of wetting agent (see Note 5) to ensure a thorough separation of materials finer than $75 \mu\text{m}$ from the coarser particles. The contents of the container shall be agitated vigorously taking care not to splash out any material, and then the wash water shall be immediately decanted over the nest of two sieves consisting of a 1.18 mm sieve superimposed on a $75 \mu\text{m}$ sieve. The use of a hand, a metal spoon, or a spatula to stir and agitate the aggregate in the wash water has been found to be satisfactory (see Note 6).

Note 5: Wetting agents such as Calgon or Alconox may be pre-mixed with water at 50 g/L. 100 ml of this solution is then added to the first wash of each test. It has been found advantageous to allow the first wash to soak for 5 min after the addition of the wetting agent and preliminary agitation.

Note 6: The agitation shall be sufficiently vigorous to completely separate the particles finer than 75 µm from the coarser particles and bring them into suspension, in order that they may be removed by decanting the wash water. Care shall be taken to avoid, as much as possible, decanting of the coarse particles of the sample. The operation shall be repeated until the wash water is clear.

4.6.2 Return all of the material retained on the nested sieves by flushing into the washed sample in the drying pan. The washed aggregate shall be dried to a constant mass at a temperature of 110 ± 5°C and the mass determined to the nearest 0.1 g (W_7) (see Note 4 above).

4.6.3 Sieve Analysis: Transfer cooled aggregate into the nest of coarse and fine sieves. Agitate on a mechanical sieve shaker. A satisfactory end point in sieving is considered to have been reached when an additional 1 min of sieving by hand fails to change the mass on any sieve by more than 1%. This is usually about 12 min. On completion of the sieving operation, separate the sieves. Each sieve shall be placed within a round stainless steel bowl and shaken manually, checking to see if additional material passes through. Any additional material should be transferred to the next smaller sieve size. When emptying the sieves, gently strike the rim of the inverted sieve with the wooden handle of the sieve brush. This helps to dislodge aggregate particles from the cloth. Individual particles stuck in coarser sieves may also require help. Following this, brush the bottom surface of the sieve cloth with a circular motion. In no case shall the particles be turned or manipulated through the sieve by hand. Weigh and record cumulatively (e.g. each successive size is to be added to the previous total) on the appropriate work sheet, the mass of the fraction retained on each sieve.

4.6.4 CALCULATION OF AGGREGATE FRACTIONS:

Calculate the per cent passing on each sieve in the test portion as follows:

$$\% \text{ passing on sieve } x = 100 - \frac{W_x \times 100}{W_3}$$

Where: W_x = cumulative mass retained on that sieve

W_3 = total mass of dry aggregate

If the aggregate breakdown procedure described under 4.2 CALIBRATION (which provides amendment to Clause 9.8 of ASTM) has been completed, a correction factor for each sieve must be determined by comparing the average gradation of the blank samples (which were not subjected to burning in the furnace) on that sieve to the average gradation of the burnt calibration samples.

4.7 TEST METHOD B (Sections 11, 12, and 13) is deleted in its entirety.

5. EXAMPLE CALCULATIONS

5.1 MOISTURE CONTENT:

Per cent moisture in sample of mix:

Mass of sample plus moisture (M_a) 1000.3 g

Mass of dried sample (M_d) 998.1 g

$$\% \text{ Moisture (Z)} = \frac{1000.3 - 998.1}{998.1} \times 100 = 0.22\%$$

5.2 ASPHALT CEMENT CONTENT:

i) If the field sample used for ignition is dry, the % AC_{by ignition} (the final value on ignition tape) will be correct.

ii) If a) of Section 4.4.1 is used, and % AC_{by ignition} (the final value on ignition tape) = 5.66%,

$$\% \text{ AC}_{\text{correct}} = \% \text{ AC}_{\text{by ignition}} - \% \text{ Moisture}$$

$$= 5.66\% - 0.22\%$$

$$= 5.44\%$$

iii) If b) of Section 4.4.1 is used, the % AC_{by ignition} (on tape) = % AC_{correct}.

5.3 AGGREGATE FRACTIONS:

Total dry mass of aggregate (W_6) 962.8 g

$$\% \text{ passing 9.5 mm sieve} = 100 - \frac{213.3}{962.8} \times 100 = 77.8$$

$$\% \text{ passing 600 } \mu\text{m sieve} = 100 - \frac{689.4}{962.8} \times 100 = 28.4$$

5.4 SWING BACK GRADING (Optional): This is the grading of the coarse aggregate (plus 4.75 mm sieve fraction) when either fraction is considered to be the total sample. For the fine aggregate, the calculation is as follows:

5.4.1 The per cent passing the 4.75 mm sieve in the total grading, divided into the per cent passing each subsequent sieve, and the result multiplied by 100 will give the fine aggregate gradation expressed as a per cent passing each sieve.

5.4.2 Example:

Given for the full gradation:

% passing the 4.75 mm sieve is 52.1

% passing the 2.36 mm sieve is 40.9

% passing the 1.18 mm sieve is 33.0

Then for the fine gradation:

$$\% \text{ passing the 2.36 mm sieve} = \frac{40.9}{52.1} \times 100 = 78.5$$

$$\% \text{ passing the 1.18 mm sieve} = \frac{33.0}{52.1} \times 100 = 63.3$$

5.4.3 To "swing back" the grading of the coarse aggregate fraction of the extracted aggregate, the following procedure is used. The per cent retained on the 4.75 mm sieve in the total grading is divided into the per cent retained on each of the coarse aggregate sieves in the total gradation and the result is multiplied by 100, to give the coarse aggregate grading expressed as the per cent retained on each sieve.

5.4.4 Example:

Given for the full gradation:

% passing the 13.2 mm sieve is 92.5

% passing the 9.5 mm sieve is 75.4

% passing the 4.75 mm sieve is 52.4

Then for the coarse gradation

$$\% \text{ ret'd on the 13.2 mm sieve} = \frac{100-92.5}{100-52.4} \times 100 = 15.8$$

$$\% \text{ ret'd on the 9.5 mm sieve} = \frac{100-75.4}{100-52.4} \times 100 = 51.7$$

6. REPORTING OF RESULTS

6.1 The report shall include the following:

6.1.1 Identification of aggregate and mix type and job mix formula,

6.1.2 Calibration temperature for a convection furnace, or the burn profile setting for a direct irradiation furnace, and data using original materials and including mean calibration factor/lab mix,

6.1.3 Testing date, laboratory log test number, and physical condition of test sample,

6.1.4 Under which subsection of Section 4.5.3 testing was done, and the per cent moisture determination if it was used,

6.1.5 Internal mass of test sample (nearest 0.1 g) before and after testing (via tape),

6.1.6 Ignition furnace end point setting of tests (1.0 g or 0.2% by mass @ 500 g sample size; 1.0 g or 0.07% @ 1500 g; 1.0 g or 0.05% @ 2000 g),

6.1.7 One test sample result measuring asphalt content (nearest 0.01%) along with test data,

6.1.8 The cumulative mass and the cumulative percentages passing for each sieve shall be reported to one decimal place. As well, the cumulative percentage may be recorded as a graph on the appropriate semi-log grading chart.

6.2 Where the residual aggregate sample requires splitting after ignition to determine gradation and two tests are carried out, the cumulative mass and the cumulative percentages passing each sieve shall be calculated as follows.

Example:

	Portion A	Portion B
Total Dry mass of Aggregate (W ₆)	962.7 g	968.3 g
Cumulative Dry Mass on 9.5 mm Sieve	213.3 g	214.4 g
$\% \text{ passing the 9.5 mm sieve} = 100 - \frac{213.3 + 214.4}{962.7 + 968.3} \times 100$		
$= 79.0$		

7. GENERAL NOTES

7.1 Aggregates in the pans are weighed while still warm to the touch to avoid absorption of moisture.

7.2 Extreme care must be exercised when transferring aggregates from 1 container to another to avoid any loss of particles.

7.3 Prior to using, check each sieve for the condition of the mesh, the soldered edges, and that the sieve nest is assembled as required in descending sizes.

7.4 All balances or load cells should be calibrated at the start of the construction season. When an operator questions the accuracy/condition of the balance, it should be recalibrated and/or inspected immediately.

7.5 All equipment and testing for LS-292 shall be located in one laboratory building only.

7.6 In northern Ontario, gneiss/migmatite aggregate was found to physically break down when burned in an ignition furnace. Aggregate gradations changed appreciably after burning, however, no aggregate loss or burnoff occurred. The larger size aggregate would crumble when squeezed by hand. Aggregate gradation corrections could not be performed since the intensity of sieving determined the extent of aggregate breakdown. Lowering the burn temperature to the minimum specified in this test method did not correct the breakdown problem. Therefore, for asphalt containing this type of aggregate, it is not possible to use the ignition oven for the determination of gradation from bituminous paving mixtures. The ignition furnace can still be used to determine the AC content.

8.0 WITHIN LABORATORY PRECISION

In a study involving 93 different laboratories, testing 2 specimens of 2 materials, the following statistical variations were obtained:

For AC%, Sample 1 Mean AC 5.10%, Std. Dev. 0.09

Sample 2 Mean AC 5.09%, Std. Dev. 0.10

Therefore, when 2 laboratories test samples of the same material, the difference between the results should not exceed 0.283% 19 times in 20.