METHOD OF TEST FOR QUANTITATIVE EXTRACTION OF ASPHALT CEMENT AND ANALYSIS OF EXTRACTED AGGREGATE FROM BITUMINOUS PAVING MIXTURES

LS-282 R30
ASTM D2172-11

4. APPARATUS

4.1 BALANCES: Of sufficient capacity and sensitivity for miscellaneous weighing, with associated accessories such as balance pans.

4.2 DRYING PANS: Seamless enamel, stainless steel, or aluminum pans with a minimum of 10 square mm horizontal surface area per gram of sample, shall be used for drying the extracted aggregate as well as the washed aggregate.

4.3 CENTRIFUGE EXTRACTOR: Either electrically or hand operated, complete with 1000 g minimum capacity bowl. For the electrically operated extractor, it is necessary to have a controlling device that will allow the speed to be set at 1800 rpm.

Note 1: Rotarex extractors have been found to be suitable for this test. Larger test sample sizes may be too large for some extractors and therefore require the sample to be done in 2 portions. Due to the nature of the solvent used, this equipment must be installed in a properly ventilated location (e.g. a fume hood or equivalent).

4.4 DRAIN TUBE: Neoprene, preferably with a 150 mm long glass insert.

4.5 FILTER PADS: Smooth, heavy weight, medium fast, low ash, qualitative type paper – E & D Grade 627 or equivalent.

4.6 MISCELLANEOUS ITEMS: 0.5 L container, 150 mm spike, 80 mm x 12 mm stainless steel spatula, stiff brush, a rubber spatula and a 75 mm wide blade scraper.

4.7 TORQUE WRENCH: 9.5 mm drive, calibrated in N-m with a capacity of approximately 22 N-m.

4.8 EXTRACTOR CONTAINERS: Any suitable containers which will permit the quantitative transfer of extract and mineral fines to the high-speed centrifuge.

4.9 TACHOMETER: A strobe type tachometer capable of determining rotational speeds in the range of 1,000 to 10,000 rpm.

4.10 GRADUATED CYLINDER: 500 ml capacity to measure the rate of flow of the extract during the recovery of mineral finds. Use 2000 ml cylinders if they are being used as receivers for the total volume of extract.

4.11 HIGH-SPEED CENTRIFUGE: As described in ASTM D2172, Centrifuge Method.

4.12 RIFFLE SPLITTER: For splitting hot mix samples. Recommended width of the individual chutes is approximately 38 mm for all types of paving mix.

5. SAMPLE SIZE

5.1 The size of the test sample shall be governed by the “Designated Large Sieve” size of the mix/mix type and shall conform to the requirements as shown in Table 1 (see Note 2).
Table 1: Size of Test Sample

<table>
<thead>
<tr>
<th>Mix Type</th>
<th>Designated Large Sieve Size (mm)</th>
<th>Minimum Mass of Sample (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HL 2</td>
<td>2.36</td>
<td>0.5</td>
</tr>
<tr>
<td>SMA 9.5, Superpave 9.5</td>
<td>4.75</td>
<td>1.0</td>
</tr>
<tr>
<td>HL 1, HL 3, HL 3A, DFC, OFC, SMA 12.5, Superpave 12.5, 12.5 FC1, 12.5 FC2</td>
<td>9.5</td>
<td>1.5</td>
</tr>
<tr>
<td>HL 4 (Binder and Surface)</td>
<td>13.2</td>
<td>1.5</td>
</tr>
<tr>
<td>Superpave 19.0, SMA 19.0</td>
<td>12.5</td>
<td>2.0</td>
</tr>
<tr>
<td>HL 8, MDBC, HDBC</td>
<td>16.0</td>
<td>2.0</td>
</tr>
<tr>
<td>Superpave 25.0</td>
<td>19.0</td>
<td>3.0</td>
</tr>
<tr>
<td>Superpave 37.5</td>
<td>25.0</td>
<td>4.0</td>
</tr>
</tbody>
</table>

Note 2: The Designated Large Sieve is a sieve size specifically designated for testing purposes.

5.2 For moisture content, a minimum sample size of 1000 g is required for all types of paving mix.

6. SAMPLE PREPARATION FROM LOOSE PAVING MIXTURES

6.1 Obtain loose field samples in accordance with specified procedures.

Plate or loose mix samples shall be reduced to the proper size.

2 methods of reducing the field sample to the testing size are acceptable: quartering or splitting using a riffle splitter.

6.1.1 Quartering: Warm the field sample to achieve sufficient workability for quartering. A conventional oven maintained at 110°C or a microwave oven (see Note 3) 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 4).

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 3: Caution: Frequent mixing may be necessary to prevent localized overheating when using a microwave oven to heat paving mixtures.

Also, the presence of metal 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 4: If required, “Pam” or equivalent used in minimal quantities has been found suitable in preventing the sample from adhering to the surface.
6.1.2 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 3 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 4 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. SAMPLE PREPARATION FROM COMPACTED PAVING MIXTURES
7.1 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 which 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 when the depth trimmed shall be approximately 4.75 mm. Dry the trimmed core to constant mass.

8. DETERMINATION OF MOISTURE CONTENT
8.1 In extraction test calculations, asphalt cement content is obtained by subtracting the extracted aggregate mass from the original mass of the test sample. If moisture is present in the original sample, it will appear as asphalt cement in the final calculation unless a correction is made. This is done by determining the amount of moisture in separate 1000 g portion and adjusting the mass of the extraction test portion or, if time permits, heating the extraction test portion to a constant mass.

The first method has the advantage that the 2 procedures can be run concurrently. Either method is acceptable.

8.2 Weigh the test portion obtained for moisture content determination and record the mass to 0.1 g. Place the test portion in an oven maintained at 110 ± 5°C and dry to a constant mass. Note 5: Constant mass is defined as no change in mass in excess of 0.1 % or 1 g, whichever is less, for each 30 minute drying period.

8.3 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

8.4 Adjust the original mass of the test portion use for extraction in accordance with Section 11.2.
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8.5 If the method of drying the original test portion is followed, then use the dried mass as the original mass in subsequent calculation.

9. EXTRACTION PROCEDURE

9.1 Measure and record the mass of the test portion to the nearest 0.1 g (W1).

9.2 Carefully transfer the mix from the balance pan to the extractor bowl keeping the funnel in place during the transfer.

9.3 Fill the 0.5 L container with the extraction solvent and wash any of the mixture remaining in the balance pan into the extractor bowl.

Put the remaining solvent into the bowl, while gently breaking down the mix with the spike or spatula.

Distribute the mix evenly around the bowl to avoid off-balance loading.

Clean all fines and asphalt off the spike or spatula with solvent and ensure all aggregate particles are covered by the solvent (see Note 6).

Note 6: For larger size bowls, increase the amount of solvent to ensure total coverage of the aggregate.

9.4 Dry the filter paper at 110°C to constant mass, to the nearest 0.1 g (Wf).

The filter paper should be weighed immediately after drying.

Filters may be pre-weighed, e.g. an established dry mass for each filter (hot) may be determined prior to using the filter.

This dry, hot mass may then be used as the “original mass of the filter” in the calculations.

After pre-drying, the filters must be kept in a dust proof container until used.

Place the filter paper, with fine side up, on the bowl.

Place the cover on the bowl (and over the filter paper) and tighten with a torque wrench. Apply a torque of 11.3 N-m to the funnel (Note 7).

Allow the sample to soak for 15 minute.

Note 7: For larger size extractor bowls, the laboratory shall have a written protocol to ensure uniform tightening of the lids.

9.5 At the end of the 15 minute soak period, with the bowl in the extractor and the cover in place, start the extractor slowly.

9.6 With the hand operated extractor, turn the handle slowly, gradually increasing the speed of the handle to 60 rpm (which is equivalent to 1800 rpm of the bowl for electrically powered extractor).

With the electrically powered extractor, start the machine slowly, allowing it to increase to a maximum of 1800 rpm (see Note 8).

Maintain this rate until the solvent ceases to flow and just drips.

Collect and retain the extract in the extract containers.

Note 8: Operate larger size extractors in accordance with manufacturer’s instructions.
9.7 Stop the extractor and remove the small cover over the funnel (if applicable). 
Pour 0.5 L of solvent through the funnel into the bowl (see Note 6 above) and rotate the bowl back and forth by hand to dislodge any material adhering to the side of the bowl.
Replace the funnel cover, allow to soak for 10 minutes, and repeat centrifuging as described in Section 9.6.

9.8 Repeat this procedure, reducing the soaking period to 5 minutes (see Note 9).
Be sure that the solution coming through the drain tube is a light straw colour.
Spin until the dripping ceases and continue for an additional 2 minutes thus ensuring a fairly dry aggregate sample.

Note 9: RHM, RAP, and samples containing slag or absorptive aggregate may require a 15 minute soak period for each repeat of the procedure.

9.9 The extract container may now contain the total extract and washings from each Rotarex cycle to be used for determination of mineral fines in the extract in accordance with Section 10.

9.10 Remove the extractor bowl and set it on a suitable stand under adequate ventilation.
Carefully remove the cover leaving the filter in place.
Tap the filter surface all around with the metal spatula so that any loose material drops into the bowl.
Lift the filter pad from the bow and carefully invert the filter over a drying pan to avoid loss of fine.
Brush or scrape the fines from the filter into the drying pan.
Loosen the aggregate from the sides of the extractor bowl with a rubber spatula (while keeping the bowl tilted) and using a stiff brush, transfer the remainder of the material from the bowl into the drying pan.
Any fines adhering to the bowl must be brushed into the drying pan.
Any fines adhering to the brush must also be transferred to the drying pan.
Bend the filter pad into a tent shape and place it on the drying pan.
Place the drying pan in the oven.
Dry to a constant mass at 110 ± 5 °C taking care not to char or burn the filter pad.

9.11 Provided low ash filter rings are used, an alternative procedure for the determination of mineral fines in the filter ring may be used (see Section 9.17).

9.12 At the end of the drying period remove the pan from the oven.
Note 10: Because dry aggregate absorbs moisture when exposed to air, determine the mass of the extracted aggregate immediately after cooling to 90-100°C to avoid damage to the balance.
A dial thermometer tared with the weighing pan has been found to be useful in determining when a suitable weigh temperature is reached.

9.13 Immediately after drying, determine the mass of the pad to the nearest 0.1 g and record. This mass is the total of the original tared mass plus the mass of the fines trapped in the filter fibres.
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9.14 Calculate the mineral fines in the filter as follows: ..............................................................
Mass of mineral fines in filter, \( W_3 = M_{ff} - M_f \)
Where:
\( M_{ff} = \) Mass of filter after extraction, g
\( M_f = \) Mass of filter, g

9.15 Immediately after drying, carefully transfer the hot aggregate from the drying pan into the balance pan, brushing any fines adhering to the drying pan into the balance pan. Determine the mass of the dried aggregate (\( M_{ba} \)), while still warm, to 0.1 g and record (see Note 10 above).............................................

9.16 Calculate the mass of dried aggregate as follows: ..............................................................
Mass of dried aggregate, \( W_4 = M_{ba} - M_b \)
Where:
\( M_{ba} = \) Mass of balance pan plus dried aggregate, g
\( M_b = \) Mass of balance pan, g

9.17 If low ash (ash residue not greater than 0.2 %) filter pads are used, dry to constant mass as required by Section 9.15, then carefully fold the filter pad and stand it on the aggregate in the weighing pan. Burn the pad by igniting it with a match or Bunsen burner.................................................................
Determine the mass of the dried aggregate and filter pad mineral fines (\( M_{ba+f} \)).................................

9.18 Calculate the mass of the dried aggregate plus filter pad mineral fines as follows: .....................
Mass of dried aggregate plus filter pad mineral fines, \( W_3 + W_4 = M_{ba+f} - M_b \)
Where:
\( M_{ba+f} = \) Mass of balance pan, dried aggregate and filter pad mineral fines, g
\( M_b = \) Mass of balance pan, g

9.19 As an alternate to the Rotarex extraction method, the reflux method can be utilized. Refer to ASTM D2172, for a complete description of this method.................................................................

10. DETERMINATION OF MINERAL FINES IN EXTRACT
10.1 This procedure uses a high speed centrifuge as described in ASTM D2172..............................
10.2 Transfer extract from containers to high speed centrifuge receiving funnel and correct for fines.................................................................
The high speed should be operated as per manufacturer’s instructions.............................................
10.3 Calculate mineral fines in the extract as follows: ..............................................................
Mass of mineral fines in extract, \( W_s = M_{cf} - M_c \)
Where:
\( M_{cf} = \) Mass of cup plus fines, g
\( M_c = \) Mass of cup, g
11 ASPHALT CEMENT CONTENT CALCULATION

11.1 Calculate the asphalt cement content in the test portion to 2 decimal places making use of appropriate work sheets.

11.2 If moisture is present in the mix and the sample has not been dried to constant mass to eliminate moisture, adjust the original mass of the mixture for the test portion or test increments as follows:

\[
\text{Dry mass of mixture, } (W_2) = \frac{W_1}{(100 + Z)} \times 100
\]

Where:
- \( W_1 \) = Original mass of test portion, g
- \( Z \) = Moisture content, %

11.3 For the test portion calculate the total mass of the dry aggregate as follows:

\[
\text{Total mass of dry aggregate, } (W_6) = W_3 + W_4 + W_5
\]

Where:
- \( W_3 \) = Mass of mineral fines on the filter
- \( W_4 \) = Mass of dry aggregates
- \( W_5 \) = Mass of mineral fines in the extract

11.4 The mass of the asphalt cement can be determined for the test portion as follows:

\[
\text{Mass of asphalt cement, } (W_7) = W_2 - W_6
\]

Where:
- \( W_2 \) = Dry mass of mixture, g
- \( W_6 \) = Dry mass of aggregate, g

11.5 The asphalt cement content expressed as a percentage of the moisture free mix is calculated as follows:

\[
\text{Asphalt cement content, } \% \ (W_8) = \frac{W_7}{W_2} \times 100
\]

11.6 If an extraction test is carried out in 2 portions, the results shall not be averaged but shall be combined to calculate a single asphalt cement content.
12 ANALYSIS OF EXTRACTED AGGREGATE

12.1 WASHING OF AGGREGATE: After the mass (W4) 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 11) to ensure a thorough separation of materials finer than 75 µm 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 2 sieves consisting of a 1.18 mm sieve superimposed on a 75 µm 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 12).

Note: 11 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 minutes after the addition of the wetting agent and preliminary agitation. It has been found advantageous to allow the first wash to soak for 5 minutes after the addition of the wetting agent and preliminary agitation.

Note 12: 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 course particles of the sample. The operation shall be repeated until the wash water is clear.

12.2 Return all 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 (W6) (see Note 10 above).
12.3 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 minute of sieving by hand fails to change the mass on any sieve by more than 1%. This is usually about 12 minutes.
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 should 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.

12.4 CALCULATION OF AGGREGATE FRACTION: Calculate the percent passing on each sieve in the test portion as follows:

\[
\text{% passing on sieve } n = 100 - \frac{W_n}{W_6} \times 100
\]

Where:

- \( W_n \) = Cumulative mass retained on that sieve
- \( W_6 \) = Total mass of dry aggregate

12.5 When the extraction test is done in 2 portions, the gradation analysis shall be performed on the total sample.

15 GENERAL NOTES

15.1 When testing cold mix, the sample must be dried at 110 ± 5°C to a constant mass to remove all solvents before testing.

15.2 Aggregates in the pans and SMM cups are weighed while still warm to avoid absorption of moisture. In order to avoid undue variation in this process it is recommended that on removal from the drying oven all samples be weighed at 90 to 100°C.

15.3 Extreme care must be exercised when transferring aggregates from one container to another to avoid any loss of particles.
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15.4 Prior to using, check each sieve for the condition of the mesh, the soldered edges, and the nest for correct order of descending sizes.

15.5 Calibrate the rpm of the centrifuge extractor and high speed centrifuge using a strobe tachometer at least once per construction season.

15.6 All balances should be calibrated at least once per year, at the start of the construction season.

15.7 If some asphalt remains in the aggregate after the 3rd washing, repeat the extraction process until the solution is a light straw colour.

15.8 The extractor bowl cover must be fastened to the bowl tightly enough to minimize the loss of fines. Uniform torquing of the bowl lids will prevent varying loss of the fines.

15.9 All extractor bowls and covers must be matched to ensure a tight fit and permanently mark to prevent mismatching.

15.10 Ensure that the funnel is in place in the bowl when the mix is being transferred from the balance pan to the bowl in order to prevent clogging of the threads with material.

15.11 Non-uniform colour and texture of the flow patterns, which continue beyond the inner rim of the extractor bowl on the filter indicate an excess loss of fines.

15.12 All equipment and testing for LS-282 including DETERMINATION OF MINERAL FINES IN EXTRACT (Section 10) shall be located in 1 laboratory building only.

COMMENTS