

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

LS-282 R33

ASTM D2172/D2172M – 17e1

3. MATERIALS

3.1 Trichloroethylene: Reagent grade chemical or grade of sufficiently high purity to permit use without lessening the accuracy of the determination.....

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. Wider blades have been used successfully for quartering (1.6 x 100 x 400 mm).....

4.7 TORQUE WRENCH: 9.5 mm drive, calibrated in N-m with a capacity of approximately 22 N-m.. This can be used either with a 19.1 mm socket with two slots milled out of the sides to fit over the steel rod in the extractor unit funnel, or with a 9.5 mm drive socket brazed into the middle of the funnel.

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

The containers should have a capacity sufficient to accommodate the volume of extract from each Rotarex cycle, and should not contaminate the specimen.

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 Extraction 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)

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Table 1: Size of Test Sample

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

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.....

Two 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.....

Motor oils must not be used for this purpose.....

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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 (see Section 8) to remove moisture..... _____

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 two 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. _____

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..... _____

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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 pad at 110°C to constant mass, to the nearest 0.1 g (M_f)..... _____

The filter pad should be weighed immediately after drying..... _____

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

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

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

Place the filter pad, with fine side up, on the bowl over the sample..... _____

Place the cover on the bowl (and over the filter pad) 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 minutes..... _____

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..... _____

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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 should 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 pad surface all around with the metal spatula so that any loose material drops into the bowl..... _____

Lift the filter pad from the bowl and carefully invert the filter pad over a drying pan to avoid loss of fines..... _____

Brush or scrape the fines from the filter pad into the drying pan..... _____

And bend the filter pad into a tent shape and place it on 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..... _____

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 pads are used, an alternative procedure for the determination of mineral fines in the filter pad 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 fibres of the filter pad _____

9.14 Calculate the mineral fines in the filter pad as follows: _____

Mass of mineral fines in filter pad, (W₃) = M_{ff} – M_f

where:

M_{ff} = Mass of filter pad after extraction, g

M_f = Mass of filter pad, 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)..... _____

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9.16 Calculate the mass of dried aggregate as follows:

Mass of dried aggregate, (W4) = $M_{ba} - M_b$

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_{baf}).....

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, ($W_3 + W_4$) = $M_{baf} - M_b$

where:

M_{baf} = Mass of balance pan, dried aggregate and filter pad mineral fines, g

M_b = Mass of balance pan, g

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_5) = $M_{cf} - M_c$

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: _____

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, %

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11.3 For the test portion calculate the total mass of the dry aggregate as follows:
 Total mass of dry aggregate, (W6) = W3 + W4 + W5

where:

W3 = Mass of mineral fines on the filter pad

W4 = Mass of dry aggregates

W5 = Mass of mineral fines in the extract

11.4 The mass of the asphalt cement can be determined for the test portion as follows:
 Mass of asphalt cement, (W7) = W2 – W6

where:

W2 = Dry mass of mixture, g

W6 = Dry mass of aggregate, g

11.5 The asphalt cement content expressed as a percentage of the moisture free mix is calculated as follows:
 Asphalt cement content, % (W8) = $\frac{W7}{W2} \times 100$

11.6 If an extraction test is carried out in two 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.....
 100ml of this solution is then added to the first wash of each test.....

1 drop of liquid dishwashing detergent in the first soak has been found to be a suitable alternative.....

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 coarse particles of the sample.....

The operation shall be repeated until the wash water is clear.....

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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 (W₉) (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.....

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.....

Ensure that all material trapped in full-sized openings of the sieve are cleaned out and included in the mass retained for that particular sieve.....

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:

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

where:

W_n = Cumulative mass retained on that sieve

W₆ = 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.....

14. REPORTING

14.1 The results will be based on a single test and shall be reported as follows:

14.1.1 The asphalt cement content (W₈) shall be reported as a percentage of the total mass of the mix to two decimal places.....

14.1.2 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.....

14.1.3 The swing back gradings, if required, shall be recorded as percentage retained or passing for each sieve, accurate to one decimal place, and shall be reported as described in Section 13.4.....

14.2 Where the test sample requires splitting to satisfy minimum mass requirements and two tests are carried out, results will be based on the sums of the values from the two test portions or increments, using two bowls. Results shall be reported with the following changes in calculations:

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- 14.2.1 The asphalt cement content shall be expressed as the cumulative percentage content for the two test increments, to two decimal places, see Example in LS clause 14.2.2..... _____
- 14.2.3 The cumulative mass and the cumulative percentages passing each sieve shall be calculated to one decimal place, see Example in LS clause 14.2.4..... _____

15 GENERAL NOTES

- 15.1 When testing cold mix, the sample must be dried at $110 \pm 5^{\circ}\text{C}$ 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..... _____
- 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..... _____
In order to tighten the cover on the extractor bowl with a torque wrench, the extractor funnel units have been modified..... _____
- Marks should be made (using a hacksaw, paint, etc.) on the steel rod to ensure that the torque wrench is centered..... _____
- 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 pad indicate an excess loss of fines..... _____
Check for a tight fit of the cover. Burrs on bowl or cover can cause a poor fit..... _____
Do not strike the machined surface of the bowl when using the brush to dislodge particles of aggregate sticking to its surface..... _____
- Overfilling the bowl with solvent can cause a loss of fines through the air vents..... _____
- 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