

Quantitative Extraction of Asphalt Binder from Asphalt Mixtures

ASTM D2172/D2172M - 17e1

4. Significance and Use

4.1 All of these test methods can be used for quantitative determinations of asphalt binder in asphalt mixtures and pavement samples for specification acceptance, service evaluation, control, and research. Each method prescribes the solvent or solvents and any other reagents that can be used in the test method.

NOTE 2—Further testing of the asphalt mixture may be performed by using sieve analysis on the extracted aggregate, Test Method D5444, or recovering the extracted asphalt binder from solution by Test Method D1856, Practice D5404/D5404M, or Practice D7906 for asphalt binder property testing. When recovering the asphalt binder for property testing, all mineral matter should be removed from the effluent.

NOTE 3—The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of Specification D3666 are generally considered capable of competent and objective testing/sampling/inspection, etc. Users of this standard are cautioned that compliance with Specification D3666 alone does not completely ensure reliable results. Reliable results depend on many factors; following the suggestions of Specification D3666 or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.

5. Apparatus

5.1 Oven, capable of maintaining the temperature at 110 ± 5 °C [230 ± 9 °F]
5.2 Pan, large enough that the asphalt mixture can be spread out in a thin layer over the bottom of the
pan
5.3 <i>Balance,</i> readable to 0.1 g, and capable of measuring the mass of sample and container. The balance
shall conform to the requirement of Guide D4753, Class GP2
5.4 Analytical Balance, readable to 0.001 g and capable of measuring the mass of the sample and
container
5.5 <i>Electric Hot Plate,</i> thermostatically controlled, of sufficient dimensions and heat capacity to permit
evaporating or refluxing of the solvent
5.6 Small-Mouth Graduate Container, 1000- or 2000-mL capacity. Optional small-mouth graduate, 100-
mL capacity
5.7 Ignition Dish, 125-mL capacity
5.8 Desiccator, a container with a lid of sufficient size to hold the ignition dish on a perforated drying
rack above the top level of the desiccant. The lid should form a good seal around the top of the
container so that air movement between the container and the atmosphere is prevented

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests...... Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.....



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7. Hazards

7.1 **Warning**—The solvents listed in Section 6 should be used only under a hood or with an effective surface exhaust system in a well-ventilated area, since they are toxic to various degrees...... Consult the current Threshold Limit Concentration Committee of the American Conference of Governmental Industrial Hygienists for the current threshold limit values.....

8. Sampling

8.1 Obtain samples in accordance with Practice D979/D979M.
8.2 Preparation of Test Specimens:
8.2.1 Separate sample by hand spatula or trowel, then split and reduce sample to required testing size in accordance to AASHTO R47.
If sample is not able to be separated or split, place sample in a large, flat pan and warm to 110 ± 5 °C [230 ± 9 °F], only heating the mixture until it is pliable enough to separate.
Split or quarter the material until the mass of material required for test is obtained and determine the mass of the sample, W1.
NOTE 5—In some cases, polymer modified mixtures need to be warmed at temperatures higher than 110 °C [230 °F] in order to split or quarter the mix. In all cases, the minimum temperature for the minimum time needed to split the mixture should be used so that any aging to the asphalt binder is minimized.

8.2.2 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 (Note 6) NOTE 6—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 masses of each increment combined before calculating the asphalt binder content (Section 14). 8.2.3 If the sample was obtained from the field and contains moisture, oven dry the HMA sample to a constant mass at a temperature of 110 ± 5 °C [230 ± 9 °F] or determine the moisture content of the sample according to Test Method D1461, so that the measured mass loss can be corrected for



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moisture	
Record mass loss as W2	
Constant mass is defined as less than 0.05 % loss in mass between consecutive 15-min intervals	

9. Test Method A – Centrifuge Extraction

9.1 Apparatus:

9.1.1 In addition to the apparatus listed in Section 5, the following apparatus is required for
Test Method A:
9.1.1.1 Extraction Apparatus, of appropriate size to revolve a bowl of approximate dimensions as shown
in Figs. 1 and 2 of the ASTM, which can be controlled by the apparatus at variable speeds up to 3600
r/min
The speed may be controlled manually or with a preset speed control
The apparatus should be provided with a container for catching the effluent thrown from the bowl and a
drain for removing the effluent
The apparatus shall be installed in a hood or an effective surface exhaust system to provide
ventilation
9.1.1.2 Filter Rings, felt or paper, to fit the rim of the bowl
9.1.1.3 Low-ash paper filter rings may be used in place of the felt filter ring (9.1.1.2)
Such filter rings shall consist of low-ash filter paper stock approximately 1.3 mm thick

The nominal base weight of the paper shall be 150 ± 14 kg $(330 \pm 30$ lb) for a 500-sheet ream with sheet size approximately 635 by 965 mm (25 by 38 in.) The ash content of the paper shall not exceed 0.2 % (approximately 0.034 g per ring)

in BEE 1 bize of bampie			
Nominal Maximum		Minimum Mass	
Aggregate Size Standard,	Sieve Size	of Sample,	
mm		kg	
4.75	(No. 4)	0.5	
9.5	3⁄8 in.	1	
12.5	1⁄2 in.	1.5	
19.0	3⁄4 in.	2	
25.0	1 in.	3	
37.5	11⁄2 in.	4	

TABLE 1 Size of Sample

9.2 Procedure:



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Dry and determine the mass of the filter ring and fit it around the edge of the bowl
Position lid on the bowl and tighten setscrew. Clamp the cover on the bowl tightly and place a beaker under the drain to collect the effluent
9.2.5 Start the centrifuge revolving slowly and gradually increase the speed to a maximum of 3600 r/min or until solvent ceases to flow from the drain
Allow the machine to stop, add 200 mL of solvent and repeat the procedure
Use sufficient solvent additions (not less than three) so that the extract is not darker than a light straw
color
Collect the effluent and the washings in a graduate container
NOTE 7—Additions of solvent greater than 200 mL may be used as appropriate for the size of the sample.
9.2.6 Drying Procedures:
9.2.6.1 Remove lid from centrifuge bowl, leaving bowl, sample and filter in place
Allow the extracted aggregate to air dry for 15 to 30 min in the ventilated hood
Place bowl, filter ring, and extracted sample into an exhaust oven at 110 <u>+</u> 5 °C [230 <u>+</u> 9 °F] for 1 to 2 h to evaporate remaining solvent
Cool bowl, filter ring, and extracted aggregate and if felt filter rings are used, brush off mineral matter adhering to the surface of the ring and add to the extracted aggregate
The mass of the extracted aggregate, W_3 , is equal to the mass of the aggregate in the bowl plus the increase in mass of the filter rings. Report mass measurements to the nearest 0.1 g
9.2.6.2 Use the following alternative procedure when low-ash filter rings are used:
Place the aggregate and filter rings in a clean metal pan
Dry as specified above
Carefully fold the dried filter ring and stand it on the aggregate
Burn the filter ring by igniting with a Bunsen burner or match
Determine the mass of the extracted aggregate in the pan, W_3 . Report mass measurements to the nearest 0.1 g
9.2.6.3 Since dry aggregate absorbs moisture when exposed to air containing moisture, determine the mass of the extracted aggregate immediately after cooling to a suitable temperature
9.2.7 Determine the amount of mineral matter in the extract by any of the test methods in Section 13.
9.2.8 Calculate the asphalt binder content as described in Section 14

10. Test Method B – Reflux Extractor



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10.1.1.3 Cylindrical Metal Frames, one or two
The lower frame shall have legs of sufficient length to support the frame, including the apex of the metal
cone and paper cone liner above the solvent level
When two frames are used, the upper frame shall have legs of sufficient length to support the metal
cone and paper cone liner at or above the top rim of the lower frame
The legs of the upper frame shall fit securely in the top rim of the lower frame
A bail handle may be provided on the inside of the top rim of each frame for convenient handling
The metal used in fabricating the frames shall be essentially inactive to the solvents used in the test method
10.1.1.4 <i>Condenser,</i> fabricated with a truncated hemispherical condensing surface and a truncated
conical top. Other suitable geometric shapes may also be used provided they accomplish the condensing
and flow functions intended
The material used in fabricating the condenser shall be essentially unreactive to water and to the
solvent used and shall be provided with suitable water inlet and outlet
10.1.1.5 <i>Filter Paper</i> , medium grade, fast-filtering
The diameter of the paper shall be such that when folded in accordance with the directions given below,
it shall completely line the metal cones in the frames (see Fig. 4 of the ASTM)
10.1.1.6 <i>Heat Resistant-Coated Wire Mesh,</i> approximately 3 mm [0.1 in.] thick for use as insulation
between the glass jar and hot plate
10.1.1.7 <i>Electric Hot Plate,</i> thermostatically controlled, of sufficient dimensions and heat capacity to
permit refluxing of the solvent as described in 10.2.2.5.
10.2 Procedure:
10.2.1 Prepare a test portion for moisture determination and extraction in accordance with the
procedure described in Section 8
10.2.2 Extraction:
10.2.2.1 Dry and determine the mass of one sheet of filter paper for each frame to be used
Fold each paper on its diameter, fold the ends over, and spread it open to form a proper size to fit inside
the metal cones
10.2.2.2 Determine the mass of each frame with its filter paper liner to the nearest 0.5 g. Record the
mass, identifying each frame by number
10.2.2.3 Place the test portion in the frame or frames
If two frames are used, distribute the test portion approximately equally between the two
The top of the test portion must be below the upper edge of the paper liner
Determine the mass of each loaded frame separately to the nearest 0.5 g. Again, record the mass
10.2.2.4 TCE, nPB, or toluene solvent can be used with this extraction method
10.2.2.5 Pour the solvent into the glass cylinder and place the bottom frame into it
The solvent level should be below the apex of the one in the (lower) frame
If two frames are used, place the upper frame in the lower frame, fitting its legs into the holes in the
upper rim of the lower frame
NOTE 8—Sufficiently denatured ethyl alcohol may be poured over the test portion(s) to wet the filter
paper.



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10.2.2.6 Place the thermal insulating pad on the hot plate and then the cylinder on the pad
Cover the condenser
Circulate a gentle, steady stream of cool water through the condenser
Adjust the temperature of the hot plate so that the solvent will boil gently and a steady stream of condensed solvent flows into the cone
If necessary, adjust the temperature of the hot plate to maintain the solvent stream at a rate necessary to keep the test portions in the cone(s) completely covered with condensed solvent
Take care not to allow condensed solvent to overflow the filter cone(s)
Continue the refluxing until the solvent flowing from the lower cone is light straw color (when viewed against a white background)
At this point, turn off the hot plate and allow the apparatus to cool with the water running in the condenser
When boiling has ceased and the cylinder is cool enough to handle, turn off the condenser and remove from the cylinder
10.2.2.7 Remove the frame assembly from the cylinder. Allow to dry in air (hood) and dry to constant mass in an oven at 110 ± 5 °C [230 ± 9 °F]
10.2.3 Determine the amount of mineral matter in the extract by any of the test methods in Section 13.
10.2.4 Calculate the asphalt binder content as described in Section 14

11. Test Method C – Vacuum Extractor

11.1 Apparatus:
11.1.1 In addition to the apparatus listed in Section 5, the following apparatus is required for Test
Method C:
11.1.1.1 Vacuum Extractor, complete with vacuum pump, gasket, rubber tubing, filter paper, support
plate, and funnel ring, similar to that shown in Fig. 5 of the ASTM
11.1.1.2 Filter Paper, medium grade, fast-filtering, 330 mm [13 in.] in diameter
11.1.1.3 Stainless Steel Beaker, having a capacity of approximately 8 L
11.1.1.4 Erlenmeyer Flasks, glass, two, having a capacity of 4000 mL each
11.1.1.5 Erlenmeyer Flask, glass, having a capacity of 1000 mL
11.1.1.6 Graduate, glass, having a capacity of 500 mL
11.1.1.7 Dial Thermometer, having a range from 10 to 80 °C [50 to 180 °F]
11.1.1.8 Watch Glass, having a 100-mm [4-in.] diameter
11.1.2 Miscellaneous Equipment—Wash bottle, large mixing spatula, stiff-bristled brush, and metal
tongs
11.2 Procedure:
11.2.1 Prepare the sample and determine the moisture content of the material in accordance with
Section 8
11.2.2 TCE, nPB, toluene, or methylene chloride solvent can be used with this extraction method.
NOTE 9—Denatured ethyl alcohol can be used to facilitate the filtering of the asphalt sample.



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11.2.6.5 Dry the extracted aggregate and filter to a constant mass in an oven at 110 \pm 5 °C [230 \pm 9 °F]
11.2.6.6 Determine the mass of the filter and aggregate in the pan and record Subtract the mass of the filter and pan to determine the mass of the extracted aggregate 11.2.7 Determine the amount of mineral matter in the extract by any of the test methods in Section 13.
11.2.8 Calculate the asphalt binder content as described in Section 14
12. Test Method D – Extraction Kettle
12.1 Apparatus:

12.1.1 In addition to the apparatus listed in Section 5, the following apparatus is required for Test Method D:
12.1.1.1 <i>Extraction Apparatus (</i> Fig. 6 of the ASTM <i>),</i> consisting of an extraction kettle of metal or borosilicate glass, fitted with a perforated basket and a condenser top
The underside of the condenser shall be covered with numerous rounded knobs to distribute the condensed solvent uniformly over the surface of the sample
The suspension of the basket shall be arranged to support the basket 13 mm [0.5 in.] above the bottom of the kettle, for immersion of test portion in the solvent, and at least 75 mm [3 in.] above the bottom of the kettle for refuming (see Note 9)
the kettle for refluxing (see Note 8)
12.2 Procedure:
12.2.1 Prepare test portions for moisture determination and extraction in accordance with the procedure described in Section 8
12.2.2 <i>Moisture</i> —Determine the moisture content of the mixtures in accordance with the test method
described in Section 8
12.2.3 <i>Extraction:</i> 12.2.3.1 Insert a filter sack in the extraction basket and determine the mass with the tare pan to
determine the total tare weight
Place the test portion (Note 6) in the filter sack and determine the total mass
Calculate the mass of the test portion
12.2.3.2 Attach the suspension rod to the loaded basket and set the assembly into the extraction kettle
Pour approximately 600 mL of solvent (6.3, 6.4, or 6.7) over the test portion. Set the condenser cover in place on the kettle
Provide a flow of cold water through the condenser lid
Raise the basket to immersion level, for example 13 mm [0.5 in.] above the bottom of kettle, by inserting the support pin through the upper hole of the suspension rod
Place the extractor on the hot plate and adjust the heating rate so that solvent is maintained at a gentle boil, avoiding vigorous boiling which might wash fines over sides of basket
12.2.3.3 Continue heating with the test portion in immersion position for 15 to 30 min and then raise the basket to refluxing level



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Increase the heat and maintain active boiling until solvent dripping from the basket appears light straw color when viewed against a white background
If a stainless steel kettle is used, lift out the basket and the condenser cover assembly for examination of
the solvent
12.2.3.4 Remove the extractor from the hot plate and allow to cool for several minutes
Lift out the basket and condenser assembly
Cover the kettle, remove the filter sack, distribute its contents onto the tared pan in which the mass of
the test portion was originally determined
Place the filter sack on top of the recovered aggregate. Dry on a steam bath and then in an oven at $110 \pm$
5 °C [230 <u>+</u> 9 °F] to constant mass
Transfer the extraction to a 1000-mL graduate. Wash the extractor clean with solvent and add the
washings to the extract
12.2.3.5 Determine the mineral matter in the extract in accordance with any of the procedures in
Section 13
12.2.3.6 Calculate the percent asphalt binder in the test portion in accordance with the procedure
described in Section 14

13. Determination of Mineral Matter

where:

G = ash in aliquot, g, V_1 = total volume, mL, and V_2 = volume after removing aliquot, mL.

13.3 Centrifuge Method:
13.3.1 For this test method use any suitable high-speed (3000 g or higher) centrifuge of the continuous-
flow type



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13.3.2 Determine the mass of a clean empty centrifuge cup (or bowl) to 0.01 g and place in the centrifuge
Position a container at the appropriate spout to catch the effluent from the centrifuging operation
13.3.3 Transfer all of the extract to an appropriate (feed) container suitably equipped with a feed control (valve or clamp, etc.)
To ensure quantitative transfer of the extract to the feed container, the receptacle containing the
extract should be washed several times with small amounts of clean solvent and the washings added to the feed container
Start the centrifuge and allow to reach a constant operational speed (for example, 9000 r/min for the SMM type and 20 000+ r /min for the Sharples type)
13.3.4 Open the feed line and feed the extract into the centrifuge at a rate of 100 to 150 mL/min
After all the extract has passed through the centrifuge, wash the feed mechanism (with centrifuge still
running) with several increments of clean solvent, allowing each increment to run through the
centrifuge until the effluent is essentially colorless
13.3.5 Allow the centrifuge to stop and remove the cup (or bowl)
Clean the outside with fresh solvent
Allow the residual solvent to evaporate in a hood and then dry the container in an oven controlled at
110 <u>+</u> 5 °C [230 <u>+</u> 9 °F]
Cool the container and again determine the mass immediately to the nearest 0.01 g
The increase in mass is the mass of mineral matter, W_4 , in the extract
13.4 Volumetric Method:
13.4.1 Place the extract in a previously tared and calibrated flask
Place the flask in a controlled-temperature bath controlled to 60.1 °C [0.2 °F], and allow the contents to
reach the temperature at which the flask was calibrated
When the desired temperature has been reached, fill the flask with solvent which has been kept at the
same temperature. Bring the level of the liquid in the flask up to the neck, insert the stopper, making
sure the liquid overflows the capillary, and remove from the bath
Wipe the flask dry, determine the mass to the nearest 0.1 g, and record this mass as the mass of flask
plus extract
13.4.2 If a controlled-temperature water bath is not used, measure the temperature of the extract and
make necessary corrections to the volume of the flask and density of asphalt and the solvent



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13.4.3 Calculate the volume of asphalt and fines in the extract as follows $V_1 = V_2 - (M_1 - M_2)$ (G1)	(2)
where: V_1 = volume of asphalt and fines in the extract, V_2 = volume of the flask, M_1 = mass of the contents of the flask, M_2 = mass of the asphalt and fines in the extract = mass of the total same extracted aggregate, and G_1 = specific gravity of the solvent determined to the nearest 0.001 in a D2111.	
13.4.4 Calculate the mass of fines in the extract as follows:	(3)
where: M_3 = mass of fines in the extract, G_2 = specific gravity of fines as determined in accordance with Test Method C128, G_3 = specific gravity of asphalts as determined in accordance with Test Method D70, $K = \frac{G_2}{G_2 - G_3}$, V_1 = as given in 13.4.3, and M_2 = as given in 13.4.3.	
14. Calculation of Asphalt Binder Content 14.1 Calculate the percent asphalt binder content in the test portion as a Asphalt binder content, $\% = [((W_1 - W_2) - (W_3 + W_4))/(W_1 - W_2)] \times 100^{-10}$	

where:

 W_1 = mass of test portion,

 W_2 = mass of water in the test portion,

 W_3 = mass of the extracted mineral aggregate, and

 W_4 = mass of the mineral matter in the extract.

NOTE 11—When ashless filter rings are not used, add the increase in mass of the felt filter ring to W_4 .

Comments