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VACUUM EXTRACTION OF BITUMEN IN PAVING MIXTURES

SCOPE

This test method is for determining the bitumen content of asphalt paving mixtures by the use of the vacuum extractor. The aggregate remaining after extraction may be used for sieve analysis. The apparatus and procedure are different than those used in the Central Laboratory Test Method 624, but the test results are similar.

PROCEDURE

A. Apparatus

1. Vacuum extractor, complete with vacuum pump, gasket, tubing, filter paper, support plate and funnel ring
2. Filter paper, medium grade, fast filtering of the diameter required to fit inside the funnel ring
3. Oven for drying
4. Glass or plastic beakers (2) graduated, at least 800-ml capacity
5. Plastic wash bottle
6. Thermometer, to at least 180°F (82°C)
7. Drying and weighing pans
8. Spatula
9. Stiff, bristle brush, approximately 1-in. (25 mm) wide (such as a close-cropped paint brush)
10. Balance with at least 3000 grams capacity and accurate to 0.5 gram
11. Sample container suitable for mixing

B. Reagents

1. Chlorinated or biodegradable solvent
2. Methyl alcohol

C. Precautions

1. Wear rubber gloves and eye protection when handling solvents.

2. Adequate ventilation must be provided.

3. Avoid inhalation of vapors.

4. Vent the exhaust from the vacuum pump into the exhaust hood or to the outside.

5. Chlorinated solvents in the presence of heat and moisture may form acids that are extremely corrosive to aluminum. Proper precautions must be taken to not allow the solvent to remain in small quantities in the tank of the vacuum extractor.

   NOTE 1: A vacuum trap attached in place of the sight tube (located to the right of the drain) can be used to collect the extract. This will prevent any solvent from entering the tank of the vacuum extractor.

D. Sample Preparation

1. Obtain and transfer into a sample container about 2000 grams of material by following the procedure described in IM 357, Preparation of Hot Mix Asphalt (HMA) Mix Samples for Test Specimens.

2. Milled asphaltic concrete paving test samples are obtained by splitting as described in IM 336, Reducing Aggregate Field Samples to Test Samples.

3. Place the milled test sample in a large shallow pan having dimensions of about 17 in. x 16 in. x 2½ in. (425 mm x 400 mm x 65 mm) and dry in the oven at about 275°F (135°C). The drying time should be only long enough to remove the moisture. After 30 minutes drying time the sample should be weighed and returned to the oven. Continue weighing every 30 minutes until sample reaches a constant weight.

4. Transfer the dried milled test sample to the sample container.

5. Allow the sample, mix test sample or the milled test sample, to cool to at least 130°F (54°C). Weigh to the nearest 0.5 gram.

6. Add solvent to the sample until it is just covered and allow to soak for at least 20 minutes.

E. Test Procedure

1. Place a dry, tared filter paper on the support plate, which is centered on the vacuum extractor. Position the funnel ring on the extractor and tighten the wing nuts finger tight.

2. Stir the sample and solvent with the spatula. Decant the asphalt solvent solution from the sample container by gently pouring it on to the filter paper.

3. Start the vacuum pump and continue vacuuming until all the solution has disappeared from the filter paper.

4. Add solvent to the sample until it is just covered, and then repeat step 2 and 3. Repeat this procedure until the solution in the sight tube of the extractor is light straw color.

   NOTE: An ultrasonic cleaner may be used to agitate the sample and solvent in place of stirring it with a spatula. In no case shall the sample and solvent be reheated.

5. After the last washing, pour the extracted aggregate into the extractor. Use the wash bottle containing solvent and thoroughly rinse all aggregate particles from the sample container and spatula into the extractor. Carefully distribute the aggregate evenly over the filter paper with the spatula.

6. Rinse the aggregate with solvent by pouring a small amount over the aggregate and then vacuum it off.

7. Continue vacuuming for approximately 10 minutes to aid in drying. To further speed drying a heat lamp may also be used, by directing it on the sample during this vacuuming time period.

8. With the spatula transfer the aggregate from the outer 1 1/2 inch of the filter towards the center.

9. Remove the funnel ring and brush any clinging aggregate either into the drying pan or on the filter. Pick up the filter paper and aggregate carefully and transfer into the drying pan.

10. Dry the aggregate and filter paper to a constant weight in an oven maintained at 275±9°F (135±5°C).

11. Drain the filtrate from the tank of the vacuum extractor. (See C2.)

12. Remove the drying pan from the oven, cool, transfer the contents including the filter paper to a weighing pan previously tared, and weigh to the nearest 0.5 grams.

13. Brush the clinging aggregate from the filter paper into the weighing pan and save the extracted aggregate for sieve analysis (IM 331).

F. Alternate Test Procedure (to be used only for samples that are difficult to extract)

1. Add solvent to the sample and allow to soak for at least 20 minutes.

2. Stir the sample with the spatula and decant the asphalt solvent solution into a separate container.

3. Repeat steps F1 and F2 two or more times or until the asphalt solvent solution begins to appear straw-colored.

4. Seal the container, containing the decanted asphalt solvent solution, with aluminum foil and allow to stand undisturbed for a minimum of 2 hours. (Allowing the fine material to settle out of this solution will facilitate the filtering process).

5. Add solvent to the remaining sample containing the coarser aggregate portion, cover with aluminum foil, and set aside with material obtained in section F4.

6. After the settling period stir the coarse sample (F5) and decant the liquid onto the filter in the vacuum extractor. Start the vacuum pump and continue vacuuming until all the solution has disappeared from the filter paper. Add a small amount of solvent to the remaining aggregate and continue this process until the liquid appears straw-colored. Set the cleaned aggregate sample aside.

7. Clean the filter with alternate washing of solvent and methyl alcohol.

8. Decant the liquid from the container described in F4 onto the vacuum filter, being careful not to disturb the settled fines, and clean the filter as described in F7.

9. Liquefy the remaining fines from F8 by adding a small amount of methyl alcohol and stirring with a spatula. Wash all particles from the container into the extractor and clean the filter again as described in F7.

10. Carefully pour the coarser aggregate portion (set-aside in step 6) onto the extractor. Using a wash bottle containing solvent, carefully wash all particles from the sample container onto the extractor. Distribute the aggregate evenly over the filter with the spatula and clean the filter with alternate washings of solvent and methyl alcohol.

11. Complete the extraction by following the procedure from E7 through E12.

    NOTE 2: After step F8 the filter paper can be replaced. If replaced, both filter papers must be combined with the extracted aggregate for drying. Be sure to subtract the weight of both filter papers in the calculations.

G. Calculations

1. Calculate the percentage of extracted bitumen to the nearest 0.1 percent as follows:

$$\text{Percent Asphalt, } B = \frac{S - A}{S} x100$$

Where: B = Percent asphalt in the mix
S = Initial weight of the test sample, grams
A = Weight of extracted aggregate, grams

H. Example

1. See example in Materials IM 331, Mechanical Analysis of Extracted Aggregate.

I. Determination of Percent Asphalt Retained (Retention Factor)

1. The percent asphalt retained shall be determined on mixtures of known asphalt content by the extraction procedure described in this method. The retention factor shall be determined for each asphalt aggregate combination with the results available when the project begins. Asphalt used to prepare the laboratory samples shall be obtained from the same source that supplies the project. If the mixture or aggregate combination is changed, the retention factor must be redetermined.

2. A mixing bowl, a large spoon and an asphalt-pouring container are needed in addition to the apparatus listed. A mechanical mixer may be used in lieu of the mixing bowl and large spoon.

3. Select, by splitting or quartering a representative portion of the uncoated aggregate sample, (usually obtained from the stockpile or cold feeds) weighing not less than 3000 grams.

4. Dry the aggregate sample either overnight or to a constant weight in an oven at 275±9°F (135±5°C).

5. Allow the sample to cool to room temperature and weigh to the nearest 0.5 grams.

6. Transfer the weighed aggregate into the mixing bowl and place into an oven maintained at 275±9°F (135±5°C); at the same time place the pouring container of asphalt in the oven. Heat the aggregate and asphalt for a minimum of 2 hours to insure the mixing temperature is attained.

7. Remove the aggregate and asphalt from the oven and immediately combine the two by weighing, to the nearest 0.5 grams, the required amount of asphalt into the aggregate. (Record the combined weight of aggregate obtained in H5 and the required weight of asphalt obtained in this step.)

8. Mix the aggregate and asphalt until complete coating is achieved. Place it in the oven at 275±9°F (135±5°C) for 1 1/2 hours. (To avoid losing part of the sample, leave the spoon or paddle in the bowl while it is in the oven.)

9. Remove the mixture from the oven and separate into two parts (use solvent and wash all remaining mixture in the bowl into one of the portions of the sample). Perform the extraction in two steps and combine the extracted aggregate weights for calculation.

10. The percent asphalt retained is the difference between the extracted asphalt content and the percent added to the aggregate in Step G.

11. **Example:** The recommended asphalt content or the intended asphalt content for the project is 6.00%.

Determination of grams of asphalt for the mixture:

Known: 1. Asphalt content required is 6.00%.

2. Weight of aggregate sample is 3093 grams.

Solve for grams of asphalt, which is x.

$$(100)\frac{\text{Weight of Asphalt}}{\text{Weight of Asphalt} + \text{Weight of Aggregate}} = 6.00\%$$

or

$$100\,\frac{x}{x + 3093} = 6.00$$

$$\begin{aligned} 100x &= 6.00\,(x + 3093) \\ 100x &= 6.00x + 18558 \\ 94x &= 18558 \\ x &= 197.4 \\ x &= 197.4 \text{ grams of asphalt} \end{aligned}$$

$$\text{Prove :}\quad (100)\frac{197.4}{197.4 + 3093} = 6.00\%$$

Laboratory Number	1	2
Known Asphalt Content	6%	6%

1. Tare Weight - Filter paper (grams)	5		5		
2. Tare Weight – Pan	171.5		171.5		
3. Tare Weight - Add Items 1 and 2	176.5		176.5		
4. Weight of Mix Sample	1679.0	+	1611.4	=	3290.4
5. Weight of Aggregate + Tare	1763.0		1695.5		
6. Weight of Aggregate (Subtract Item 3 from Item 5)	1586.5	+	1519.0	=	3105.5
7. Weight of Extracted Asphalt Subtract Item 6 from Item 4)					184.9

8. Percent Extracted Asphalt
   (Item 7 divided by Item 4) 5.6%
   8a. Percent Asphalt Retained
   (Retention Factor)
   8b. Percent Asphalt
   (Add Item 8 and 8A)

Difference between known asphalt content and extraction result:

1. 6.0 - 5.6 = 0.4
   = 0.4 Percent Asphalt Retention

Figure 1. Vacuum Extractor
(Pump, Tank, Support Plant and Funnel Ring)