

EXTRACTION OF BINDER FROM ASPHALT PAVING MIXTURES

1. SCOPE: This test method covers the determination of the asphalt binder content (AC) of asphalt mixtures by use of a centrifuge extractor (Method A) or a vacuum extractor (Method B).
2. SIGNIFICANCE AND USE:

- 2.1. This method is one of a group of approved means of determining the AC of asphalt mixtures. Other means include Kentucky Method (KM) 64-436, *Asphalt Binder Content Determination of Asphalt Mixtures by Plant Recordation*, KM 64-437, *Determination of Asphalt Binder Content of Asphalt Mixtures Using the Nuclear Asphalt Content Gauge*, KM 64-438, *Asphalt Binder Content Determination of Asphalt Mixtures Based on the Maximum Specific Gravity*, or AASHTO T 308, *Determining the Asphalt Binder Content of Hot-Mix Asphalt (HMA) by the Ignition Method*.
- 2.2. Use this method for process-control or acceptance testing. The Department of Highways will use this method for informational testing.

NOTE 1: Utilize the solvent of choice. However, this method does not purport to address any safety or disposal considerations associated with its use. Determine and implement appropriate safety and health practices, handling and storage procedures, disposal limitations, and comply with any applicable regulatory limits prior to use.

Ensure the solvent is effective in removing all asphalt binder from the aggregate. When testing the extracted aggregate further, ensure the solvent leaves the sample “film-free” after water rinsing, if applicable.

CAUTION: Ascertain the suitability of the solvent for use in the particular type of extractor available. Some solvents may not be suitable in all types of extractors.

- 2.3. Use AASHTO T 164, *Quantitative Extraction of Bitumen from Bituminous Paving Mixtures*, Method A or B, when performing “referee” testing or dispute resolution.
3. SAFETY PRECAUTIONS:
 - 3.1. All solvents are potentially toxic and/or hazardous; use these materials under an exhaust hood and/or in an area with adequate ventilation. Avoid inhaling the vapors and ~~avoid~~ skin contact. If skin contact occurs, rinse with soap and water. For vacuum extractors, vent the exhaust from the vacuum pump outside, and drain the vacuum extractor daily. Avoid open flames or any other potential source of ignition.

NOTE 2: Some possibility of spontaneous combustion exists when using “biodegradable” or organic solvents. Handle and store the solvent according to the directions specified on

the materials safety data sheet (MSDS). Strictly observe any precautions or handling practices indicated by the manufacturer. Properly dispose of any cleaning items, such as rags.

3.2. Use the following operator safety equipment: eye protection, apron, and gloves.

4. APPARATUS:

4.1. Provide a ventilation system, capable of removing fumes and dust, conforming to Section 401 of the *Standard Specifications for Road and Bridge Construction*.

4.2. Provide scales capable of weighing 5,000 g to an accuracy of 1.0 g.

4.3. Provide an extraction solvent.

4.4. Provide an ultrasonic cleaner and insert tray or bowl (optional).

4.5. Provide a hot plate or oven.

4.6. Provide water (if applicable).

4.7. Provide a propane burner (optional).

NOTE 3: Consider Methods A and B, without dust-loss corrections, to be rapid, field-test methods only. Before using either method, determine that the procedure is satisfactory by comparing the results to a “referee” method (such as AASHTO T 164). See Subsection 8.4 for the procedure for determining dust loss through the vacuum extractor (Method B).

4.8. For the centrifuge method (Method A) only:

4.8.1. Provide a centrifuge in which the bowl may be revolved at controlled, variable speeds up to 3600 revolutions per minute (rpm).

4.8.2. Provide a filter-ring pad (capable of retaining aggregate particles larger than 6 microns), medium grade.

4.8.3. Provide the necessary miscellaneous equipment, such as a measuring cup or can, scoop with a rectangular cross-section, thermometer, spatula, brushes, large drying pans, etc.

4.9. For the vacuum-extractor method (Method B) only:

4.9.1. Provide a vacuum extractor and vacuum pump, assembled as shown in Figure 1.

4.9.2. Provide a watch glass.

4.9.3. Provide filter paper (capable of retaining aggregate particles larger than 6 microns), 13-in. diameter, medium grade.

- 4.9.4. Provide a filtering aid (Celite). Ensure that all of the material is finer than a No. 200 sieve, or else determine and apply a correction.
- 4.9.5. Provide the necessary miscellaneous equipment, such as a bowl, spoons, thermometer, putty knife, drying pan, wash bottle, propane burner (optional), etc.
5. SAMPLES: Refer to KM 64-425, *Sampling Asphalt Mixtures*, for procedures for obtaining and reducing samples to the required test-portion size.
6. GENERAL PROCEDURE (Centrifuge - Method A):
- 6.1. Check the scales to ensure its preparedness for use (zeroed, balanced, etc.).
- 6.2. Inspect the centrifuge bowl for warping. If the bowl is acceptable, record the tare weight of the empty and clean centrifuge bowl as W_1 . Weigh, and record the weight of, the filter pad(s).
- NOTE 4: The scale may be zeroed (tared) with the bowl on the scale. In this case, add the test portion to the bowl, and record the weight as W_2 .
- ~~6.3.~~ ~~6.3.~~ Weigh the required test portion into the bowl, and record the total weight of the test portion and bowl as W_2 .
- 6.4. Allow the sample to cool. Pour enough solvent into the bowl to cover the mixture. Allow the solvent-covered sample to soak for a minimum of 20 minutes, while stirring or agitating occasionally.
- 6.5. Place the bowl containing the sample and solvent into the centrifuge.
6. 6. Place the filter pad(s) on top of the bowl so that the pad(s) extend(s) past the rim of the bowl for its entire circumference. Place the smoothest, tightest side of the filter down, facing the bowl. Place the lid on the bowl, and tighten it enough to secure the filter pad(s) in place.
- 6.7. Complete the tightening of the bowl lid by hand until it is adequately sealed. A tight seal is important as it minimizes the loss of the finer fractions of the aggregate. Do not tighten the lid with a wrench; this practice may cause warping of the lid.
- 6.8. Revolve the centrifuge slowly at first; then, increase the speed to a maximum of 3600 rpm. Continue this operation until solvent ceases to flow from the centrifuge drain. Collect the effluent, and allow the aggregate particles to settle. For visual inspection of dust loss, use a "see-through" container to collect the effluent. When desired, perform a visual inspection to obtain an approximate degree of dust loss. Retain the effluent from each wash for determining the amount of dust loss (see Note 3). Repeat the adding of solvent and washing until the extracted solvent is a light "straw" color. Normally, use no more than five cycles. For those mixtures from which the asphalt is difficult to extract (such as mixtures with high absorption, high asphalt contents, special additives or materials, etc.), an ultrasonic cleaner may be used to aid in the removal of the asphalt prior to placing the solvent/asphalt solution into the centrifuge for extraction.

If using an ultrasonic cleaner, determine the sample weight prior to placing the solvent into the ultrasonic cleaner. Pour cold water in the ultrasonic cleaner, and place the insert tray with the sample in the ultrasonic cleaner. If the tray floats, remove some of the water. Add sufficient solvent to just cover the sample. Turn on the ultrasonic cleaner to break the sample apart and dissolve the asphalt.

- 6.9. When using biodegradable/organic solvent, rinse the solvent film from the aggregate prior to drying the aggregate (when the manufacturer's recommendations so specify). If so, add enough clear water to cover the sample, and wash the solvent "film" completely from the aggregate. Repeat the adding of water and washing until the sample has been thoroughly washed. Again, normally use no more than five cycles. Allow the last wash to spin longer than the preceding ones, removing as much of the water as possible, thus expediting the drying of the aggregate.

NOTE 5: When not using a biodegradable/organic solvent to extract the asphalt from the mixture, do not perform the procedure given in Subsection 6.9 prior to drying the aggregate.

- 6.10. After the final wash, disengage the bowl; loosen the lid, and remove and clean the filter pad(s) by scraping and brushing the loose aggregate particles into a separate drying container. Remove the remaining aggregate particles from the centrifuge bowl, and place the particles into the drying container. Ensure no aggregate is lost while performing the test.
- 6.11. Remove all solvent and moisture from the sample prior to the final weighing. Depending on the type of solvent utilized, accomplish this process by heating the sample in an oven or on a hot plate with adequate ventilation. Determine the weight gain from the filter pad(s) by weighing the dried pad(s) and subtracting the original weight of the pad(s) from the dried weight. Record the "pad gain" as W_3 .

NOTE 6: Adequately vent solvent fumes from the drying process outside the laboratory.

6. 12. Weigh the extracted aggregate; record this weight as W_4 .
- 6.13. When determining the gradation of the aggregate according to KM 64-433, *Wet-Sieve Analysis of Aggregates Used in Asphalt Mixtures*, retain the dried aggregate.

7. CALCULATIONS AND REPORT (Centrifuge - Method A):

- 7.1. Calculate the % AC by the following formula:

$$\% AC = \frac{(W_2 - W_1) - (W_3 + W_4)}{W_2 - W_1}, \text{ where}$$

W_1 = Tare weight of the bowl (g) if applicable;

W_2 = Weight of the coated sample and the bowl (g);

W_3 = Pad(s) weight gain (g); and

W_4 = Weight of the extracted dry aggregate (g).

- 7.2. When performing acceptance testing, report the % AC to the nearest 0.1 percent on the appropriate *Asphalt Mixtures Acceptance Workbook* (AMAW).

8. GENERAL PROCEDURE (Vacuum Extractor - Method B):

NOTE 7: Equipment modification as described in Note 9 will likely be required. It is strongly recommended that a vacuum-extractor bypass be attached to allow the filtrate to be collected in separate vacuum flasks. This type of bypass permits the solvent washes to remain totally separate from the water washes (if utilizing a solvent in which the water rinses are necessary). Gel formation can occur when water contacts some biodegradable/organic solvents in particular concentration ranges. This gel, if formed, can clog filter pads and cause fouling of the equipment, as well as causing disposal difficulties. AASHTO T 164 provides a detailed sketch of a typical vacuum extractor apparatus.

- 8.1. Record the weight of the insert tray or bowl used to wash the sample. Place the required test portion in the bowl or insert tray, and record the weight of the bowl or insert tray plus the test portion.

NOTE 8: The scale may be zeroed (tared) with the insert tray or bowl on the scale. In this case, add the test portion to the insert tray or bowl, and record the weight.

- 8.2. Place a dry filter of known weight on the vacuum extractor, taking care to center the filter on the funnel wing, and tighten the wing-nuts “finger tight.”
- 8.3. Weigh 50 g of filtering aid (Celite) in a quart container. Add approximately 500 ml of solvent. Stir to form a slurry. Pour the slurry over the filter paper, washing the quart container and spoon to ensure that all the filtering aid is transferred to the filter paper. Vacuum off the solvent until the filtering aid dries and the surface cracks. When experience indicates the mixture is “fast-filtering,” do not use the filtering aid.
- 8.4. Allow the sample to cool. Add sufficient solvent to just cover the sample. Stir the sample to break it up and dissolve the asphalt. If an ultrasonic cleaner is being used, pour cold water in the ultrasonic cleaner, and place the insert tray with the sample in the ultrasonic cleaner. If the tray floats, remove some of the water. Add sufficient solvent to just cover the sample. Turn on the ultrasonic cleaner to break up the sample and aid in the removal of the asphalt. Allow the sample to soak as described in Subsection 6.8.

NOTE 9: If it is desired to inspect the effluent for dust loss, bypass the vacuum-extractor collection tank, and catch the solvent/asphalt/aggregate fines mixture in a glass flask for viewing or dust-loss determination (see Note 3).

- 8.5. Place the watch glass on the filtering-aid surface. Pour the solvent/asphalt solution onto the watch glass carefully so as not to damage the filtering-aid surface. Start the vacuum pump to draw off the solvent/asphalt solution. Apply vacuum until the liquid has been drawn off. Add additional solvent, and repeat this process until the aggregate has been washed clean of asphalt. Pour the solution directly on the filter without using the watch glass when the filtering aid is not used. After the last solvent wash, replace the flask that has been used to collect the solvent washes, and attach a clean flask for collection of the

“water washes.”

8.6. When biodegradable/organic solvent is utilized, check the manufacturer’s recommendation regarding rinsing the solvent film from the aggregate prior to drying. If recommended, add enough water to cover the sample and stir well. The water will turn “milky-white” at this point. After completely stirring, pour the water onto the watch glass, and vacuum the water through the filter. Repeat the “water washes” until the water is clear (approximately four washes).

8.7. After pouring off the last “water wash,” lift the watch glass from the filter. Carefully clean the watch glass by washing the filtering aid and aggregate onto the filter.

NOTE 10: When not using a biodegradable/organic solvent to extract the asphalt from the mixture, ignore the instructions given in Subsections 8.6 and 8.7 prior to drying the aggregate.

8.8. Transfer the cleaned aggregate to the vacuum extractor.

8.9. Spread the cleaned aggregate over the filter paper. Cover the aggregate with water for a final wash. Vacuum off the water. Move the aggregate and filtering aid away from the sides of the vacuum extractor.

8.10. Record the weight of the drying pan. Disassemble the vacuum extractor, and place the aggregate and filtering aid, if used, in the drying pan. Clean the fines from the filter paper into the drying pan, and then place the filter paper in the drying pan.

8.11. Dry the aggregate, any filtering aid, and filter in an oven or on a hot plate. Weigh, and record the total weight of, the drying pan, dried aggregate, and filter paper (and filtering aid if used).

8.12. If the gradation of the aggregate is to be determined according to KM 64-433, retain the dried aggregate.

9. CALCULATIONS AND REPORT (Vacuum Extractor - Method B):

9.1. Determine the total weight of the test portion by subtracting the weight of the insert tray or bowl from the total weight of the insert tray plus the test portion or bowl plus the test portion, as applicable. See Subsection 8.1.

9.2. Determine the total weight of the dried aggregate by subtracting the weight of the drying pan plus the original weight of the filter paper plus 50 g of filtering aid (when used) from the gross weight as determined in Subsection 8.11.

9.3. Calculate the % AC by the following formula:

$$\% AC = 100 \left(\frac{\text{Total Weight of Test Portion} - \text{Weight of Dry Aggregate}}{\text{Total Weight of Test Portion}} \right)$$

9.4. See the attached worksheet for determining the % AC with or without filtering aid.

9.5. When performing acceptance testing, report the % AC to the nearest 0.1 percent on the appropriate AMAW.

10. DISTRICT OR DIVISION OF MATERIALS TESTING:

10.1. The Department will perform testing in either the District laboratory or the Division of Materials, for informational purposes, dispute resolution, etc., from either recently produced mixture or pavement cores, according to Subsections 3-9 (with the following exceptions):

10.2. The sample will require heating in order to obtain a test portion. Heat the sample as necessary to separate it with a spatula or trowel (so that the asphalt can be adequately removed). Do not overheat the sample such as to cause the asphalt to drain from the aggregate. When pavement samples are tested, thoroughly heat the sample in an oven at 230 ± 9 °F. After heating the sample, break down the material, and thoroughly blend the heated sample prior to obtaining a test portion.

11. EXTRACTOR CAPACITY: Ensure the size of the test portions fully complies with the provisions of KM 64-425.

12. DUST LOSS CORRECTION: When either experience or testing identifies a significant dust loss from the extractor, additional equipment and testing may be required as described in AASHTO T 164.

APPROVED _____
Director
DIVISION OF MATERIALS

DATE 3/4/0412/28/04

Kentucky Method 64-405-~~0405~~
Revised 3/4/0412/28/04
Supersedes KM 64-405-~~0304~~
Dated 1/13/033/4/04

Attachment

km4050-054.doc

CALCULATION WORKSHEET

ASPHALT CONTENT DETERMINATION BY VACUUM EXTRACTION

With Filtering Aid

Weight of insert tray or bowl plus sample (g) _____

Weight of insert tray or bowl (g) _____

Weight of sample (g) _____

Weight of drying pan and 50 g of filtering aid,
dried aggregate, and dried filter pad (g) _____

Weight of drying pan and 50 g of filtering aid,
plus original weight of filter pad (g) _____

Weight of dried aggregate (g) _____

Asphalt content (%) = $\frac{100 * (\text{Weight of sample} - \text{Weight of dried aggregate})}{\text{Weight of sample}}$ = _____

Without Filtering Aid

Weight of insert tray or bowl plus sample (g) _____

Weight of insert tray or bowl (g) _____

Weight of sample (g) _____

Weight of drying pan, dried aggregate, and
dried filter pad (g) = _____

Weight of drying pan plus original weight
of filter pad (g) = _____

Weight of dried aggregate (g) _____

Asphalt content (%) = $\frac{100 * (\text{Weight of sample} - \text{Weight of dried aggregate})}{\text{Weight of sample}}$ = _____

CALCULATION WORKSHEET

ASPHALT CONTENT DETERMINATION BY VACUUM EXTRACTION AND ACCOMPANYING EXTRACTED GRADATION

Extracted Gradation

Sieve Size	Grams Retained	Percent Retained	Percent Passing
2 in.			
1 1/2 in.			
1 in.			
3/4 in.			
1/2 in.			
3/8 in.			
No. 4			
No. 8			
No. 16			
No. 30			
No. 50			
No. 100			
No. 200			
Pan			
Total			