



**INDIANA DEPARTMENT OF TRANSPORTATION  
DIVISION OF MATERIALS AND TESTS**

**QUANTITATIVE EXTRACTION OF  
ASPHALT/BINDER AND GRADATION OF  
EXTRACTED AGGREGATE FROM HMA MIXTURES**

**ITM No. 571-21**

**1.0 SCOPE.**

- 1.1 This method of test covers the procedure for the quantitative determination of the asphalt/binder content and gradation of the extracted aggregate of HMA mixtures.
- 1.2 The HMA mixture is extracted with a suitable solvent, depending on the type of extraction apparatus used. The asphalt content is calculated by determining the difference of the weight (mass) of the HMA mixture and the extracted aggregate, fibers if used, and the fines recovered from the extracted solvent and water rinse, if required. The gradation of the extracted aggregate is then determined.
- 1.3 This ITM may involve hazardous materials, operations, and equipment and may not address all of the safety problems associated with the use of the test method. The user of the ITM is responsible for establishing appropriate safety and health practices and determining the applicability of regulatory limitations prior to use.

**2.0 REFERENCES.**

**2.1 AASHTO Standards.**

- M 231 Weighing Devices Used in the Testing of Materials
- T 30 Mechanical Analysis of Extracted Aggregate
- T 164 Quantitative Extraction of Asphalt Binder from Hot Mix Asphalt (HMA)

**2.2 ITM Standards.**

- 572 Dying HMA Mixtures
- 580 Sampling HMA
- 587 Reducing HMA Samples to Test Size

**2.3 ASTM Standards.**

- D8159 Automated Extraction of Asphalt Binder from Asphalt Mixtures

**3.0 TERMINOLOGY.** Definitions for terms and abbreviations shall be in accordance with the Department's Standard Specifications, Section 101.

**4.0 SIGNIFICANCE AND USE.** This ITM shall be used to determine the asphalt content and gradation of the extracted HMA mixture.

**5.0 APPARATUS.**

5.1 Balance, a Class G2, in accordance with AASHTO M 231

5.2 Electric skillet, with a thermostatic heat control capable of heating to 221°F

5.3 Oven, capable of maintaining the temperature at  $221 \pm 9^\circ\text{F}$

5.4 Pans and containers as needed

5.5 Sieves, in accordance with AASHTO T 30

5.6 Spatulas and trowels as needed

5.7 Stiff bristle brush, 1 in. in diameter

5.8 Thermometer, armored with a range of 100°F to 450°F, readable to 5°F

5.9 Wash bottle

5.10 Extraction apparatus

**6.0 REAGENTS.**

6.1 Alternative Extraction Solvent.

6.2 Trichloroethylene

**Note 1:** INDOT labs will use trichloroethylene exclusively

**7.0 SAFETY PRECAUTIONS.**

7.1 Provide adequate ventilation and avoid inhalation of vapor. The ventilation fan shall be operating during the testing.

7.2 The exhaust from the vacuum pump shall be vented outside.

7.3 The extraction solvent shall be an approved solvent.

**8.0 SAMPLING.** Sampling shall conform to the requirements of ITM 580.

**9.0 PREPARATION OF SAMPLE.**

- 9.1** If the sample is not sufficiently soft to separate with a spatula or a trowel, place the sample in a large flat pan and heat to a maximum of  $221 \pm 9^\circ\text{F}$  only until the sample may be handled. Separate the sample as uniformly as possible, using care not to fracture the mineral aggregate.
- 9.2** Reduce the sample in accordance with ITM 587, minimum weight procedure. The approximate minimum size of the sample shall be in accordance with the following:

<b>Mixture Designation</b>	<b>Minimum Weight (mass) of Sample, g</b>
4.75 mm	1000
9.5 mm	1500
12.5 mm	2000
19.0 mm, OG 19.0 mm	3000
25.0 mm, OG 25.0 mm	4000

- 9.3** After reduction, place the sample on a flat non-stick surface, and continue to stir the sample using a spatula. When the sample is cool enough to handle with gloves, continue to separate particles using care not to fracture the aggregate.

## **10.0 PROCEDURES.**

### **10.1 Method A - Centrifuge Extractor.**

**10.1.1** Method Specific Apparatus. In addition to the apparatus listed in 5.0, the following apparatus is required for Method A.

- a) Centrifuge Extractor having controlled variable speed up to 3600 rpm
- b) Filter rings, to fit the rim of the centrifuge bowl
- c) Continuous-Flow Filterless high speed centrifuge having the ability to reach a minimum speed of 10,000 rpm
- d) Aluminum cups for Continuous-Flow Filterless high speed centrifuge
- e) Balance conforming to the requirements of AASHTO M 231, Class G1
- f) No. 200 sieve

**10.1.2** Centrifuge Extractor.

- a) Dry the sample to constant weight in accordance with ITM 572. (This is not required if the sampled has been conditioned per Directive 303)
- b) Dry the filter to a constant mass at  $221 \pm 9^{\circ}\text{F}$
- c) Allow the sample to cool and ensure particles are separated without fracturing
- d) Place the sample in a tared extraction bowl and determine the weight
- e) Place the bowl on the extractor and cover the sample with solvent
- f) Allow the solvent to break down the sample for a minimum of 1 hour, not to exceed 3 hours (soaking period).
- g) Assemble the centrifuge extractor with a dry weighed filter paper in place. Clamp the cover on tightly and place a container under the drain to collect the extracted solvent.
- h) Start the centrifuge revolving slowly and gradually increase the speed until a steady stream of solvent flows from the drain (approx. 1400 rpm). Continue until the solvent ceases to flow from the drain.
- i) Stop the centrifuge. Add a minimum of 500 mL of solvent.
- j) Repeat the extraction and solvent addition process in 10.1.2h and 10.1.2i according to the following:

Mixture	Minimum Solvent Additions (after the soak)
Dense Graded/OG	5
SMA	8

- k) After the extracted solvent is a light straw color (when viewed against a white background) remove the extractor lid and leave the open bowl in the fume hood to allow fumes to dissipate
- l) Carefully remove the filter and inspect the sample for conglomerated material. If found, break the conglomerated material up and saturate with solvent. Reinstall the filter and clamp the bowl into the extractor for solvent additions as needed
- m) Remove the lid and allow fumes to dissipate

- n) Remove the bowl and rinse the extractor clean using the solvent
- o) Dry the extracted aggregate and filter to a constant weight in the oven or skillet at  $221 \pm 9$  °F
- p) Collect the extract for mineral matter determination.

**10.1.3 Mineral Matter Determination by Continuous-Flow Filterless high speed centrifuge**

- a) Determine the mass of an empty centrifuge cup to the nearest 0.1 g
- b) Place the cup in the filterless centrifuge and position a container to catch the effluent
- c) Pour the extract through a No. 200 sieve into the centrifuge feeder bowl and wash any retained material back into the sample.
- d) Start the filterless centrifuge and allow it to reach a constant speed
- e) Open the feed line to allow extract to be fed at a rate of 100 to 150 mL per minute
- f) The remaining extract, if any, added to the feeder bowl and container rinsed several times with solvent
- g) Rinse the feed mechanism several times with clean solvent until effluent is colorless
- h) Stop the filterless centrifuge and remove the cup
- i) Clean the outside of the cup with solvent and place cup into fume hood to allow solvent to evaporate
- j) Dry the cup in an oven at  $221 \pm 9$  °F to a constant weight
- k) Allow the cup to cool and immediately determine the mass to the nearest 0.1g
- l) Report the increase of the cups mass from the original dry mass of the cup as the mass of the mineral matter (cup fines)

**10.1.4** Calculation Using Centrifuge.

- a) The asphalt content in percent is calculated by the following formula:

$$\text{Asphalt Content, \%} = \frac{W_1 - (W_2 + W_3)}{W_1} \times 100$$

where:

$W_1$  = weight of sample, g

$W_2$  = weight of extracted aggregate, g

$W_3$  = weight of fines in extracted solvent (cup and filter fines), g

**10.2 Method B - Vacuum Extractor.**

**Note 2:** It should not be assumed that results from Method B are equivalent to Method A or C.

**10.2.1** Method Specific Apparatus. In addition to the apparatus listed in 5.0, the following apparatus is required for Method B:

- a) Vacuum extractor
- b) Filter paper, medium grade, fast filtering of the diameter required to fit the extractor, (Eaton-Dikeman #633-70)
- c) Vacuum pump
- d) Pan, round, bowl type, stainless steel
- e) No. 200 sieve

**10.2.2** Vacuum Extractor for Mixture without Fibers.

- a) Dry the sample to a constant weight in accordance with ITM 572. Determine the weight of a dry filter paper at  $221 \pm 9^\circ\text{F}$ .
- b) Weigh approximately 50 g (record exact weight) of a filtering aid, such as celite, into a 1000 mL flask, add 500 mL of extraction solvent, and swirl until the filtering aid is completely in suspension. 100 g of filtering aid may be used if the solvent does not readily pass through the filter. Immediately pour the solution onto the filter. Start the vacuum pump and let the pump run until the pad formed by the filtering aid is surface dry and begins to crack slightly. Collect the solvent which goes through the filter in a flask and pour the solvent onto the filter.

- c) If the sample is in an oven bag, remove the sample from the bag, and weigh the sample. Add enough solvent to cover the sample and stir vigorously (Note 3). Stirring shall continue until the sample is completely separated and essentially clean of the asphalt.

**Note 3:** Soaking the sample after stirring for several minutes may be beneficial in removing the asphalt from the aggregate. Extended soaking is acceptable only for aggregates with low water absorption values.

- d) Place a No. 200 sieve on the filter of the extractor and start the vacuum pump.
- e) Pour the solvent from the initial rinse onto the No. 200 sieve. If the solvent does not readily pass through the filter, lightly scrape the celite to remove the fines. After the solvent has decanted through the filter, pour approximately 500 mL of solvent into the extractor and let this decant through the filter.
- f) Add 200 - 400 mL of solvent to the sample again and decant the solvent into the extractor. Repeat this procedure until the aggregate is clean of asphalt and the extracted solvent is a light straw color (when viewed against a white background). Normally, approximately five rinses shall be needed to completely clean the sample (slag mixtures may require additional rinses.). Rinse the asphalt from the side of the extractor and the sieve with solvent.
- g) Allow the vacuum pump to run until all of the solvent has been decanted through the filter and the filter has a completely dry appearance.
- h) Gently stir the layer of celite to break the crust of fines which has formed on the pad. Caution is required to prevent tearing or puncturing the filter paper.
- i) Start the pump and pour water through the No. 200 sieve to remove any film left from the solvent.
- j) If the mixture of water and solvent forms a gel, replace the flask that has been used to collect the solvent and collect the water rinse separately.
- k) 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 through the No. 200 sieve, start the vacuum pump, and decant the water into a flask.

- l)** Repeat 10.2.2 k until the water is clear. Allow the vacuum pump to run until the filter pad is dry.
- m)** Rinse the fines accumulated on the No. 200 sieve into the extracted aggregate. Remove the filter ring and lift the filter and place the filter into another pan. Dry the filter to a constant weight in the oven or skillet at  $221 \pm 9^{\circ}\text{F}$ , and weigh immediately upon removal from the oven or skillet.
- n)** Dry the extracted aggregate to a constant weight in the oven or skillet at  $221 \pm 9^{\circ}\text{F}$ , and weigh.
- o)** The fines in the extracted solvent and water rinse shall be collected in accordance with 10.1.3.

**10.2.3** Calculation without Fibers. The asphalt content in percent is calculated by the following formula:

$$\text{Asphalt Content, \%} = \frac{W_1 - (W_2 + W_3)}{W_1} \times 100$$

where:

$W_1$  = weight of sample, g

$W_2$  = weight of extracted aggregate, g

$W_3$  = weight of fines in extracted solvent and water rinse (cup and filter fines), g

**10.2.4** Vacuum Extractor for Mixture with Fibers.

- a)** The extraction procedure shall be conducted in accordance with 10.2.2.
- b)** Rinse the fines and fibers accumulated on the No. 200 sieve into the extracted aggregate
- c)** Dry the extracted aggregate and fibers to a constant weight in the oven or skillet at  $221 \pm 9^{\circ}\text{F}$ , and weigh
- d)** Remove the filter ring, lift the filter and place the filter into a separate bowl. Dry the filter in the oven or skillet at  $221 \pm 9^{\circ}\text{F}$ , and weigh immediately upon removal from the oven or skillet.
- e)** Place the extracted aggregate and fibers into the necessary series of sieves. After shaking, the fibers shall be removed from the sieves.



- f) Place the fibers and three 1 in. washers into the series of sieves on the No. 4 sieve and shake for 10 minutes. Remove the fibers from the sieves and weigh separately.
- g) The extracted aggregate weight that is used to calculate the gradation may be determined by subtracting the weight of the fibers determined in 10.2.4 f from the combined weight of extracted aggregate and fibers in 10.2.4 c and weight of fines in 10.2.4 d.
- h) The fines in the extracted solvent and water rinse shall be collected in accordance with 10.1.3.

### 10.2.5 Calculation with Fibers.

- a) The asphalt content is calculated by the following formula:

$$\text{Asphalt Content, \%} = \frac{W_1 - (W_2 + W_3 + W_4)}{W_1} \times 100$$

where:

$W_1$  = weight of test sample, g

$W_2$  = weight of extracted aggregate, g

$W_3$  = weight of fines in extracted solvent and water rinse, g

$W_4$  = weight of fibers, g

- b) The fiber content in the mixture is calculated by the following formula:

$$\text{Fiber Content, lbm/t (kg/Mg)} = \frac{W_4}{W_1} \times 2000 (1000)$$

## 10.3 Fines Correction Factor.

**10.3.1** The extraction procedure shall be performed in accordance with 10.2.2 or 10.2.4.

**10.3.2** The fines in the extracted solvent and water rinse shall be collected in accordance with 10.1.3.

### 10.3.3 Calculation with Fines Correction.

- a) A fines correction factor shall be determined by the following formula:

$$\text{Fines Correction Factor (C)} = \frac{W_3}{W_5}$$

where:

$W_3$  = weight of fines in extracted solvent and water rinse, g

$W_5$  = weight of extracted aggregate passing the No. 200 sieve, g

- b) The fines correction factor shall be applied to each subsequent extraction test for each mixture. The correction factor is multiplied by the weight of extracted aggregate passing the No. 200 sieve, and the calculated weight is considered the fines in the extracted solvent and water rinse.
- c) The asphalt content in percent is calculated by the following formula:

$$\text{Asphalt Content, \%} = \frac{W_1 - (W_2 + (C \times W_5))}{W_1} \times 100$$

where:

C = fines correction factor

$W_1$  = weight of sample, g

$W_2$  = weight of extracted aggregate, g

$W_5$  = weight of extracted aggregate passing the No. 200 sieve, g

#### 10.4 Method C - Continuous-Flow Filterless high speed centrifuge without Extractor.

**Note 4:** It should not be assumed that results from Method C are equivalent to Method A or B.

**10.4.1 Method Specific Apparatus.** In addition to the apparatus listed in 5.0, the following apparatus is required for Method C:

- a) Pan, round, bowl type, stainless steel
- b) No. 200 sieve
- c) Suitable containers to collect the extracted solvent and water rinse
- d) Continuous-Flow Filterless high speed centrifuge having the ability to reach a minimum speed of 10,000 rpm

- e) Aluminum cups for Continuous-Flow Filterless high speed centrifuge

#### 10.4.2 With Continuous-Flow Filterless high speed centrifuge

- a) Dry the sample to a constant weight in accordance with ITM 572 (This is not required if the sampled has been conditioned for per Directive 303)
- b) Add enough extraction solvent to cover the sample and stir vigorously (Note 3). Stirring shall continue until the sample is completely separated and essentially clean of the asphalt.
- c) Place a No. 200 sieve over the container (placing the No. 200 sieve in a large funnel prior to collecting the solvent in the container is beneficial)
- d) Pour the solvent from the initial rinse through the No. 200 sieve into the container
- e) Add 200 to 400 mL of solvent to the sample and again pour the solvent through the No. 200 sieve into the container. Repeat this procedure until the aggregate is clean of asphalt. Normally, approximately five rinses shall be required to completely clean the sample (slag mixtures may require additional rinses.). Rinse the asphalt from the sieve with the solvent.
- f) Replace the container that has been used to collect the solvent, and collect the water rinse separately.
- g) Add enough water to cover the sample and stir well. The water shall turn milky-white at this point. After completely stirring, pour the water through the No. 200 sieve into the container.
- h) Repeat 10.4.2 g until the water is clear
- i) Rinse the fines accumulated on the No. 200 sieve into the extracted aggregate
- j) Dry the extracted aggregate to a constant weight in the oven or skillet at  $221 \pm 9^{\circ}\text{F}$  and weigh
- k) The fines in the extracted solvent and water rinse shall be collected as in 10.1.3, except weighing may be performed to the nearest 0.1 g. The extracted solvent and water rinse shall be poured through the Continuous-Flow Filterless high speed centrifuge. The amount

of material in the centrifuge cup(s) shall be verified to assure that the cup was not overloaded. If the cup was overloaded with fines, then an additional clean cup(s) shall be used, and the extracted solvent and water rinse shall be poured through the centrifuge again. This procedure is repeated until the cup is not overloaded.

**10.4.3** Calculation with Continuous-Flow Filterless high speed centrifuge. The asphalt content in percent is calculated by the following formula:

$$\text{Asphalt Content, \%} = \frac{W_1 - (W_2 + W_3)}{W_1} \times 100$$

where:

$W_1$  = weight of sample, g

$W_2$  = weight of extracted aggregate, g

$W_3$  = weight of fines in extracted solvent and water rinse, g

## **10.5 Method D – Automatic Extractor**

**10.5.1** Extraction using automatic extraction equipment shall be performed in accordance with ASTM D8159, except as follows:

- a)** For sampling and sample preparation, this ITM will govern.
- b)** In cases of discrepancy, this ITM will govern.

**10.5.2** A minimum of 6 wash cycles will be utilized for HMA mixtures and a minimum of 8 wash cycles will be used for SMA mixtures.

- a)** Dry cycles shall equal the number of wash cycles.

**11.0 GRADATION.** The gradation of the extracted aggregate shall be in accordance with AASHTO T 30 except that decantation through the No. 200 sieve is not required. The entire sample of extracted aggregate is tested for gradation.

**12.0 REPORT.** The asphalt content and gradation are reported to the nearest 0.01% and the fiber content is reported to the nearest 0.1 lbm/t.