# State of Nevada Department of Transportation Materials Division

# METHOD OF TEST FOR DETERMINING BITUMEN RATIO OR ASPHALT CONTENT IN HOT MIX ASPHALT BY THE IGNITION METHOD

#### **SCOPE**

This test method covers the determination of bitumen ratio or asphalt content of hot mix asphalt (HMA) paving mixtures and pavement samples by ignition of the asphalt cement in a muffle furnace at 482°C or 538°C (900°F or 1000°F).

Aggregate obtained by this method may be used for sieve analysis using Test Method Nev. T206 (Field Method) or AASHTO T 30 in their entirety except for the following changes:

Test Method Nev. T206 (Field Method)

- 1. PREPARATION OF TEST SAMPLES, delete 2.
- 2. CALCULATIONS, delete NOTE adjusting gradation for mineral filler.

# AASHTO T 30

- 1. Delete section entitled SAMPLE.
- 2. Delete first paragraph of section entitled PROCEDURE.

## REFERENCED DOCUMENTS

Test Method Nev. T200 - Standard Methods of Sampling Stone, Gravel, Sand, and Paving Mixtures for use as Highway Materials (Field Method)

Test Method Nev. T206 - Method of Test for Sieve Analysis of Coarse and Fine Aggregate (Field Method)

Test Method Nev. T306 - Method of Determining Moisture Content of Asphalt Mixtures or Mineral Aggregate using Microwave Ovens (Field Method)

AASHTO T 30 - Mechanical Analysis of Extracted Aggregate

AASHTO T 329 - Moisture Content of Hot-Mix Asphalt (HMA) by Oven Method

## SIGNIFICANCE AND USE

This method can be used for the determination of bitumen ratio or asphalt content of hot mix asphalt paving mixtures and pavement samples for quality control, specification acceptance, and mixture evaluation. Bitumen ratio is the ratio of the weight of asphalt to the dry weight of aggregate in an asphalt mixture. Asphalt content is the ratio of the weight of the asphalt to the total weight of the asphalt mixture.

Note 1: Although bitumen ratio will be used throughout this test procedure, it is also valid for determining asphalt content. However, the calibration factor will not be the same. Therefore, bitumen ratio and asphalt content cannot be interchanged. One or the other must be selected and used exclusively throughout the calibration and testing process.

## **SUMMARY OF TEST METHOD**

Two samples with a known bitumen ratio are prepared, burned by ignition, and bitumen ratios calculated. The difference between the actual and the measured bitumen ratio is calculated for each sample. The average difference is determined and recorded as a calibration factor. This calibration factor is then used to calculate a corrected bitumen ratio on HMA paving mixtures and pavement samples.

#### **APPARATUS**

- 1. Furnace Forced air ignition furnace capable of maintaining temperatures to  $538 \pm 5.5^{\circ}$ C (1000  $\pm$ 10°F), with an internal balance thermally isolated from the furnace chamber accurate to 0.1 g. The balance shall be capable of weighing a 3000 g sample in addition to the sample baskets. A data collection system will be included so that the weight can be automatically determined and displayed during the test. The furnace shall have a built-in computer program to calculate the change in mass of the sample baskets and provide for the input of a calibration factor to account for aggregate loss or incomplete asphalt combustion. The furnace shall provide a printed ticket with the initial sample mass, sample mass loss, temperature compensation, calibration factor, corrected asphalt content (%), corrected bitumen ratio (%), test time, and test temperature. The furnace chamber dimensions shall be adequate to accommodate a sample size of 3000 g. The furnace shall provide an audible alarm and visible indicator when the sample mass loss does not exceed 0.01 percent of the total sample mass for three consecutive minutes. The furnace door shall be equipped so that the door cannot be opened during the ignition test. A method for reducing furnace emissions shall be provided. If the furnace uses filters, they must be self-cleaning ceramic filters. The furnace shall be vented into a hood or to the outside and shall have no noticeable odors escaping into the laboratory. The furnace shall have a fan with the capability to pull air through the furnace to expedite the test and to reduce the escape of smoke into the laboratory.
- 2. Balance Readable to 0.1 g and capable of measuring the appropriate sample sizes. Balance must meet the requirements of AASHTO M 231, Class G2.

- 3. Sample Baskets Two or more nested sample baskets will be used that allow the samples to be thinly spread and allow air to flow up through and around the sample particles. The sample shall be completely enclosed with screen mesh or perforated steel plate with maximum and minimum openings of No. 6 (3.35 mm) and No. 30 (600 μm), respectively.
- 4. Catch Pan Sufficient size to hold the sample baskets so that aggregate particles falling through the screen mesh are caught. Wire guards will be provided to attach the sample baskets firmly to the catch pan.
- 5. Retriever Capable of safely moving the sample baskets in and out of the heated furnace.
- 6. Oven Capable of maintaining temperatures to 135°C (275°F) for drying samples.
- 7. Safety Equipment Face shield or safety glasses to provide eye protection when loading and unloading furnace. Gloves capable of withstanding temperatures up to 538°C (1000°F). A protective cage to surround the sample baskets during cooling.
- 8. Miscellaneous Equipment Pans for holding samples. Spatulas and brushes for removing asphalt mixtures and aggregate from baskets and pans.
- 9. Microwave Oven For drying samples.

## SAMPLE PREPARATION

- 1. Obtain samples of the paving mixture in accordance with Test Method Nev. T200 (Field Method).
- 2. By the method of splitting, obtain representative samples with sufficient material for the ignition procedure. The size of the test sample shall be governed by the nominal maximum aggregate size of the mixture and conform to the mass requirements of Test Method Nev. T206 (Field Method) or as listed below in Table 1. (Table 1 is for Materials Division use only).

Table 1

# Size of Sample

Nominal Maximum Aggregate Size	Minimum Mass of Sample
37.5 mm (1 1/2 in.)	4000 g
25.0 mm (1 in.)	3000 g
19.0 mm (3/4 in.)	2000 g
12.5 mm (1/2 in.)	1500 g
9.5 mm (3/8 in.)	1200 g
4.75 mm (No. 4)	1200 g

If the sample size exceeds the capacity of the ignition furnace, the sample may be split into representative portions and the results combined after testing. Sample sizes shall not be more than 500 g greater than the minimum recommended sample mass.

#### MOISTURE CONTENT

Moisture content will be determined by AASHTO T 329 or Test Method Nev. T306 (Field Method).

#### **CALIBRATION**

1. A calibration factor is required to account for the loss of aggregate during the ignition process or for the incomplete combustion of the asphalt in a sample. The calibration factor may be affected by the type, source and gradation of aggregate used, by the type, quantity, and brand of asphalt used, and by the addition of mineral filler. Therefore, to optimize accuracy, a calibration factor shall be established for every aggregate type and source and for every type and brand of asphalt used. Any change in the aforementioned will require that a new calibration factor be established. The calibration factor shall be determined by testing samples of the asphalt mixture only.

In addition to the reasons listed above, a new calibration factor will be required under the following conditions:

- a. Change in the recommended bitumen ratio of 0.4% or greater.
- b. Change in any of the aggregate bin percentages of 4.0% or greater.
- c. Change in the amount of mineral filler or a change in the method of adding mineral filler.
- d. If the Resident Engineer has a reason to suspect a material change that may affect the calibration factor.

The calibration factor is also dependent upon the testing temperature. Testing will be performed at either 482°C or 538°C (900°F or 1000°F). A test temperature is selected that will provide adequate ignition of the asphalt, while minimizing aggregate loss.

The calibration factor will be verified by testing a single sample at least once a week by the procedure outlined in this section. If the difference between the measured and actual bitumen ratio differs from the calibration factor by more than 0.20, a new calibration factor will be established as per this section.

2. The calibration factor to be used for HMA paving mixtures shall be the average difference between the measured and actual bitumen ratios of two samples tested at the same temperature to the nearest 0.01.

3. Two samples will be prepared to determine the calibration factor. Calibration samples must conform to the mass requirements of Test Method Nev. T206 (Field Method) or Table 1. For field laboratories, sample the aggregate (total blend of aggregate minus mineral filler, unless marinated, example shown below) using the automatic sampling device in accordance with Test Method Nev. T200 after the plant has been calibrated. Asphalt used for calibration purposes must be tested to ensure specification compliance. Both calibration samples shall be mixed at design bitumen ratio and at the design mineral filler content using the specified method.

Calculate the amount of mineral filler based on actual bin percentages. Use 1% mineral filler for the coarse aggregate and 2% mineral filler for the fine aggregate.

# Example:

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7% - 1 in. aggregate
                         (coarse)
21% - 3/4 in. aggregate
                         (coarse)
25% - 1/2 in. aggregate
                         (coarse)
20% - crushed fines
                         (fine)
27% - washed sand
                         (fine)
53% coarse aggregate X 0.01
                                = 0.53% mineral filler
47% fine aggregate X 0.02
                                = 0.94% mineral filler
Total mineral filler
                                = 1.47\%
(Example: 2500 g of blended aggregate including 1.47% mineral filler)
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Formula:

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<u>dry aggregate + mineral filler</u> = dry aggregate without mineral filler

1 + [(% mineral filler) ÷ 100]

<u>2500 g of aggregate</u> = 2463.8 g of aggregate without mineral filler

1.0147 mineral filler
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- 4. Preheat the ignition furnace to 538°C (1000°F).
- 5. Dry the mixture samples in accordance with the MOISTURE CONTENT section.
- 6. Test samples in accordance with the PROCEDURE section, Steps 4 through 11, using a correction factor of zero.
- 7. If the difference between the measured bitumen ratios of the two samples is less than 0.15, these results may be used to calculate a calibration factor. If the difference between the measured bitumen ratios for the two samples exceeds 0.15, repeat the calibration procedure with two additional samples. Discard the high and the low result and determine the calibration factor from the remaining two results. The difference between these two remaining results must be within the 0.15.

- 8. Calculate the difference between the measured and actual bitumen ratios for each sample. The calibration factor (CF) is the average of the measured differences to the nearest 0.01.
  - Note 2: It is possible that some asphalt/aggregate combinations will produce a measured bitumen ratio less than the actual ratio. This will produce a negative calibration factor. Rather than correcting for aggregate loss, a negative calibration factor corrects for the asphalt that is not completely ignited during the test procedure.
- 9. If the calibration factor exceeds 1.00%, lower the test temperature to 482°C (900°F) and repeat the calibration procedure. Use the calibration factor obtained at 482°C (900°F) even if it exceeds 1.00%.
  - Note 3: If excessive aggregate breakage occurs at 538°C (1000°F), the test temperature may be lowered to 482°C (900°F) and the calibration procedure repeated even if the calibration factor does not exceed 1.00%.
- 10. The calibration factor is to be recorded and used to determine the corrected bitumen ratio of HMA paving mixtures as per the PROCEDURE section. The temperature for testing HMA samples in the PROCEDURE section, shall be the same temperature selected for testing calibration samples.
- 11. A database can be kept to record the required testing temperatures for various asphalt/aggregate combinations. This database can be used to predetermine the correct testing temperature of a specific asphalt/aggregate combination for calibration purposes.

## **PROCEDURE**

- 1. Preheat the ignition furnace to the proper temperature for the sample to be tested as determined in the CALIBRATION section.
- 2. Obtain a sample of hot mix asphalt paving mixture in accordance with the SAMPLE PREPARATION section.
- 3. Oven dry the sample or determine the moisture content in accordance with the MOISTURE CONTENT section. If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in an oven at 135°C (275°F) until it can be handled.
- 4. Weigh and record the mass of the sample basket assembly sample baskets, catch pan and guards.
- 5. Evenly distribute the sample into the baskets and spread into thin layers. If basket size allows, keep the mix approximately 25 mm (1 in.) away from the sides of the baskets to reduce aggregate loss. Determine the total mass of the sample, baskets, catch pan, and guards. Calculate and record the initial mass (MI) of the sample (total mass mass of the sample basket assembly) to the nearest 0.1 g.
- 6. Program the initial mass (MI) of the sample and the calibration factor, obtained in the CALIBRATION section, into the ignition furnace controller.

- Note 4: Take care to input the correct sign (positive or negative) when entering the correction factor
- 7. Open the furnace door and place the sample baskets inside. Care must be taken to ensure that the samples baskets do not contact the furnace wall. Verify that the sample mass (including the basket(s)) displayed on the furnace scale equals the total mass recorded within ± 5 g. Differences greater than 5 g or failure of the furnace scale to stabilize may indicate that the sample basket(s) are contacting the furnace wall. Initiate the test by starting the ignition furnace. This will lock the furnace door and start the combustion blower.
  - Note 5: The furnace temperature will drop below the setpoint when the door is opened, but will recover with the door closed and when ignition occurs. Sample ignition typically increases the temperature well above the setpoint, depending on sample size and bitumen ratio.
- 8. Heat the sample in the furnace at the specified temperature until the mass loss does not exceed 0.01 percent of the sample mass for three consecutive one minute intervals. The visible indicator and audible stable alarm will indicate that the test is complete. At this point, stop the test and print out the results. (This may be an automatic function of the furnace).
  - Note 6: Mass loss may occasionally level out for a few minutes during the test causing the furnace to signal premature completion. Care must be taken to ensure that complete ignition has been obtained.
- 9. Remove the sample from the furnace and allow it to cool to room temperature. This will take approximately 30 minutes. The sample should be placed on a heat resistant surface and covered with the protective cage during cooling.
- 10. Corrected bitumen ratio will be automatically computed by the ignition furnace and reported on a printed tape. If a moisture content has been determined, subtract the moisture content from the bitumen ratio on the printed tape and report the difference as the corrected bitumen ratio.

The furnace shall calculate the corrected bitumen ratio or asphalt content by the following formulas (excluding temperature compensation):

Corrected Bitumen Ratio = 
$$\left( \left( \begin{array}{c} \underline{MI - MF} \\ MF \end{array} \right) \times 100 \right)$$
 - CF

where:

MI = Total mass of HMA sample prior to ignition.

MF = Total mass of aggregate and mineral filler (if applicable) remaining after ignition.

CF = Calibration factor obtained in accordance with the CALIBRATION section.

Corrected Asphalt Content = 
$$\left(\left(\begin{array}{c} \underline{MI - MF} \\ \underline{MI} \end{array}\right) \times 100 - CF\right)$$

where:

MI = Total mass of HMA sample prior to ignition.

MF = Total mass of aggregate and mineral filler (if applicable) remaining after ignition.

CF = Calibration factor obtained in accordance with the CALIBRATION section.

11. If for any reason the furnace cannot be used to automatically calculate a corrected bitumen ratio or asphalt content, calculations can be performed manually. Determine the total mass of the sample after ignition, baskets, catch pan, and guards. Calculate and record the final mass (MF) of the sample (total mass - mass of the sample basket assembly) to the nearest 0.1 g. Using the initial mass (MI) and final mass (MF), manually calculate a corrected bitumen ratio or asphalt content with the formulas in Step 10 of the PROCEDURE section. Apply a moisture correction if necessary.

- Note 7: Since dry aggregate absorbs moisture when exposed to air containing moisture, determine the mass of the aggregate remaining after ignition immediately after cooling to a suitable temperature.
- 12. The aggregate remaining after ignition may be used for gradation analysis if desired. The gradation will be performed using AASHTO T 30 or Test Method Nev. T206 (Field Method), in accordance with the SCOPE section.

#### **REPORT**

1. Bitumen ratio shall be reported to the nearest 0.1%. Gradation will be reported as per AASHTO T 30 or Test Method Nev. T206 (Field Method).