

**OHD L-26  
METHOD OF TEST FOR  
DETERMINATION OF BITUMEN CONTENT IN  
BITUMINOUS PAVING MIXTURES**

**METHOD A**

**1. SCOPE.**

- 1.1 This test covers the determination of asphalt content of bituminous paving mixtures by ignition of the asphalt cement at 1000°F ( 538°C) in a furnace. The asphalt in a sample of bituminous paving material is burned by ignition at 1000°F (538°C) . The asphalt content is calculated from the mass of ignited aggregate, moisture content, and temperature compensation for the change in mass of the sample container. The asphalt content is expressed as mass percentage of the moisture-free mixtures. This method may not be applicable to mixes containing fibers or ground tire rubber (dry process).
- 1.2 This method may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

**2. APPARATUS.**

- 2.1 A forced air ignition furnace, capable of maintaining the temperature at 1202°F (650°C), with an internal balance thermally isolated from the furnace chamber accurate to 0.1 g. The balance will be capable of weighing a 3,500 gram sample in addition to the sample baskets. The furnace will calculate a temperature compensation factor for the change in weight of the sample baskets and provide for the input of a correction factor for aggregate loss. The furnace will provide a printed ticket with the initial specimen weight, specimen weight loss, temperature compensation, correction factor, corrected asphalt content (%), test time, and test temperature. The sample chamber dimensions will be at least 14x10x14 inches (35x26x35 cm) (WxHxD) A method for reducing furnace emissions will be provided. The furnace will provide an audible alarm and indicator light when the sample weight loss remains constant for two consecutive minutes. The furnace door will be locked until the completion of the test procedure.

**NOTE:** The National Center for Asphalt Technology (NCAT) Asphalt Content Tester manufactured by Barnstead/Thermolyne and distributed by QC Resource (800) 296-7171 has been found to be suitable.

- 2.2 Tempered stainless steel 3/32 inch (2.36 mm) mesh or otherwise perforated baskets.
- 2.3 One stainless steel catch pan.
- 2.4 Oven capable of maintaining 257°±9°F (125±5° C).
- 2.5 Balance, 8-kg minimum capacity, sensitive to 1.0 g for weighing sample in baskets.
- 2.6 Safety Equipment: safety glasses or face shield, high temperature gloves, and long sleeve jacket. Additionally, a heat resistant surface capable of withstanding 1202°F (650°C) and a protective cage capable of surrounding the sample baskets will be provided.

2.7 Miscellaneous Equipment: pan dimensions (LxWxD) 15x15x2 inches (38x38x5 cm) minimum for transferring samples after ignition, spatulas, bowls, and wire brushes.

**3. PREPARATION OF TEST SPECIMENS.**

3.1 The bituminous mixture used for preparing the test sample will be obtained in accordance with AASHTO T 168. The test sample will be prepared by splitting or quartering this material, in accordance with AASHTO T 248, to the appropriate size as shown in section 3.3.

3.2 If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in a large flat pan and warm to 257°±9°F (125±5° C) for 25 minutes. The sample will not be heated for more than 1 hour.

3.3 The recommended size of the test sample is governed by the maximum aggregate size in the mixture and can be found in the table below:

| Maximum Aggregate Size (inches) | Maximum Aggregate Size (mm) | Recommended Mass of Sample (grams) |
|---------------------------------|-----------------------------|------------------------------------|
| 2.5                             | 63                          | 4800 ± 1200                        |
| 2                               | 50                          | 3600 ± 900                         |
| 1.5                             | 37.5                        | 2400 ± 600                         |
| 1                               | 25.0                        | 1800 ± 500                         |
| 3/4                             | 19.0                        | 1400 ± 400                         |
| 1/2                             | 2.5                         | 1000 ± 300                         |
| 3/8                             | 9.5                         | 700 ± 200                          |
| 3/16                            | 4.75                        | 500 ± 200                          |

When the mass of the test sample exceeds the capacity of the equipment used, the test sample may be divided into suitable increments for testing. The results are appropriately combined for calculation of bitumen content.

**4. MOISTURE AND VOLATILES DETERMINATION.**

4.1 Split or quarter the remaining mixture to obtain a test sample for moisture and volatile determination. The size of the test sample should be the same as recommended in Section 3.3 or 1,000 ± 250 grams. Place the test sample in a tared pan and determine the mass to the nearest 0.1 gram and record as  $W_{1m}$ . Dry to a constant mass in an oven at a temperature of 257°±9°F (125±5° C) Determine the mass of the sample to the nearest 0.1 gram and record as  $W_{5m}$ . The moisture and volatiles weights are used in the determination of bitumen content in section 7.

**NOTE:** The same test specimen may be used for the moisture and volatiles determination and bitumen determination.

5. **CALIBRATION.** Two calibration procedures are provided. Either method may be required by the engineer. For mix designs containing Recycled Asphalt Pavement (RAP) the mixture calibration procedure will be used. To compensate for the binder in the RAP the binder content of the RAP must first be determined by Method C or Method D of this test procedure. This determination is normally performed by the laboratory initially performing the mix design. Certain aggregate types may result in an unusually high correction factor and erroneous gradation results. Such mixes should be calibrated and tested at a lower temperature, typically 900°F (482°C) or as approved by the Engineer.

**5.1 MIXTURE CALIBRATION.**

- 5.1.1 This method may be affected by the type of aggregate in the mixture. Accordingly, to optimize accuracy, a calibration factor will be established with the testing of a set of calibration samples for each mix type. This procedure must be performed before any acceptance testing is completed.
- 5.1.2 Two calibration specimens conforming to the mass requirements of Section 3.3 shall be prepared at the design asphalt content  $\pm 0.5\%$ . If RAP is used, adjust the asphalt quantity used in mixing the specimens to allow for the asphalt binder contained in the RAP.
- 5.1.3 The freshly mixed specimens may be placed directly in the sample baskets. If allowed to cool, the samples must be preheated in a  $257^{\circ}\pm 9^{\circ}\text{F}$  ( $125\pm 5^{\circ}\text{C}$ ) oven for 25 minutes.
- 5.1.4 Preheat the ignition furnace to  $1000^{\circ}\text{F}$  ( $538^{\circ}\text{C}$ ) . Record the furnace temperature (set point) prior to the initiation of the test.
- 5.1.5 Enter a correction factor of 0.00 in the ignition furnace.
- 5.1.6 Weigh and record the weight of the two sample baskets and catch pan.
- 5.1.7 Place the bottom sample basket in the catch pan. Evenly distribute approximately one half of the calibration specimen in the lower basket taking care to keep the material away from the edges of the basket. Place the upper sample basket on the bottom basket assembly. Evenly distribute the remaining specimen in the top basket. Use a spatula or trowel to level the specimen.
- 5.1.8 Weigh and record the specimen, baskets, and catch pan. Calculate and record the initial weight of the sample specimen (total weight minus the weight of the sample basket assembly).
- 5.1.9 Input the initial weight of the sample specimen in whole grams into the ignition furnace controller. Verify that the correct weight has been entered.
- 5.1.10 Open the chamber door and place the sample baskets in the furnace. Close the chamber door and verify that the sample weight (including the baskets) displayed on the furnaces scale equals the total weight recorded in Section 5.1.8 within 5g. Differences greater than 5 grams or failure of the furnace scale to stabilize may indicate that the sample baskets are contacting the furnace wall. Initiate the test by pressing the start/stop button. This will lock the sample chamber and start the combustion blower.

- 5.1.11 Allow the test to continue until the stable light and audible stable indicator indicates the test is complete. Press the start/stop button. This will unlock the sample chamber and cause the printer to print out the test results.
- 5.1.12 Repeat steps 5.1.3 through 5.1.11 for the second calibration sample.
- 5.1.13 Once the calibration specimens have been burned, determine the difference between the actual and measured asphalt contents for each sample. The mix correction factor is the average of the measured differences ( $\pm 0.1\%$  of each sample).
- 5.1.14 The calibration procedure for a given mix should be repeated for every 50,000 tons (45,350 metric tons) of material produced or as requested by the engineer.

## 5.2 **AGGREGATE CALIBRATION.**

- 5.2.1 This method may be affected by the type of aggregate in the mixture. Accordingly, to optimize accuracy, a calibration factor will be established with the testing of a set of calibration samples for each mix type. This procedure must be performed before any acceptance testing is completed. This procedure should not be used if RAP is contained in the mix design.
- 5.2.2 Two blank (aggregate only) calibration specimens conforming to the mass requirements of Section 3.3 shall be prepared to meet the JMF.
- 5.2.3 Preheat a specimen in a  $257^{\circ}\text{F} \pm 9^{\circ}\text{F}$  ( $125 \pm 5^{\circ}\text{C}$ ) oven for 20 minutes. Thoroughly mix the specimen with a spatula prior to introducing it into the sample baskets.
- 5.2.4 Preheat the ignition furnace to  $1067^{\circ}\text{F}$  ( $575^{\circ}\text{C}$ ). Record the furnace temperature set point prior to the initiation of the test. Enter a correction factor of 0.0 in the ignition furnace.
- 5.2.5 Weigh and record the weight of the two sample baskets and catch pan.
- 5.2.6 Place the bottom sample basket in the catch pan. Evenly distribute approximately one half of the calibration specimen in the lower basket taking care to keep the material away from the edges of the basket. Place the upper sample basket on the bottom basket assembly. Evenly distribute the remaining specimen in the top basket. Use a spatula or trowel to level the specimen.
- 5.2.7 Weigh and record the specimen, baskets, and catch pan. Calculate and record the initial weight of the sample specimen (total weight minus the weight of the sample basket assembly).
- 5.2.8 Input the initial weight of the sample specimen in whole grams into the ignition furnace controller. Verify that the correct weight has been entered.
- 5.2.9 Open the chamber door and place the sample baskets in the furnace. Close the chamber door and verify that the sample weight (including the baskets) displayed on the furnaces scale equals the total weight recorded in Section 5.2.8 within 5g. Differences greater than 5 grams or failure of the furnace scale to stabilize may indicate that the sample baskets are contacting the furnace wall. Initiate the test by pressing the start/stop button. This will lock the sample chamber and start the combustion blower.

- 5.2.10 Allow the specimen to remain in the furnace until the stable light comes on. If the light does not come on within one hour, note percent loss and recheck every 15 minutes until no change in percent loss occurs. At this time press the start/stop button to end. This will unlock the sample chamber and cause the printer to print out the test results.
- 5.2.11 Repeat steps 5.2.3 through 5.2.11 for the second specimens.
- 5.2.12 The mix calibration factor is calculated by averaging the results obtained in 5.2.11.
- 5.2.13 The calibration procedure for a given mix should be repeated for every 50,000 tons (45,350 metric tons) of material produced or as requested by the engineer.

**6. TEST PROCEDURE.**

- 6.1 Preheat the ignition furnace to 1000°F (538°C). Record the furnace temperature (set point) prior to the initiation of the test.
- 6.2 Enter the correction factor for the specific mix to be tested as determined in Section 5 in the ignition furnace.
- 6.3 Weigh and record the weight of the two sample baskets and catch pan (with guards in place).
- 6.4 Prepare the sample as described in Section 3. Place the bottom sample basket in the catch pan. Evenly distribute approximately one half of the specimen in the lower basket taking care to keep the material away from the edges of the basket. Place the upper sample basket on the bottom basket assembly. Evenly distribute the remaining specimen in the top basket. Use a spatula or trowel to level the specimen.
- 6.5 Weigh and record the sample, baskets, and catch pan. Calculate and record the initial weight of the sample specimen (total weight minus the weight of the sample basket assembly).
- 6.6 Input the initial weight of the sample specimen in whole grams into the ignition furnace controller. Verify that the correct weight has been entered.
- 6.7 Open the chamber door and place the sample baskets in the furnace. Close the chamber door and verify that the sample weight (including the baskets) displayed on the furnaces scale equals the total weight recorded in Section 6.5 within 5g. Differences greater than 5 grams of failure of the furnace scale to stabilize may indicate that the sample baskets are contacting the furnace wall. Initiate the test by pressing the start/stop button. This will lock the sample chamber and start the combustion blower.
- 6.8 Allow the test to continue until the stable light and audible stable indicator indicate the test is complete. Press the start/stop button. This will unlock the sample chamber and cause the printer to print out the test result.
- 6.9 Open the chamber door, remove the sample baskets and allow to cool to room temperature (approximately 30 minutes). Allow the specimen to cool to room temperature in the sample baskets.
- 6.10 Empty the contents of the baskets into a flat pan. Use a small wire sieve brush to ensure that any residual fines are removed from the baskets.
- 6.11 Perform the gradation analysis according to AASHTO T 30.

7. **CALCULATIONS.**

$$\% AC = \frac{\frac{(P_{5m} \times W_{1m})}{100} - (W_{1m} - W_{5m})}{W_{5m}} \times 100$$

Where:

- $P_{5m}$  = Apparent asphalt content from ignition oven  
 $W_{1m}$  = Mass of moisture sample before drying  
 $W_{5m}$  = Mass of moisture sample after drying

**METHOD B**

1. **SCOPE.**

- 1.1 This method covers the determination of bitumen content in bituminous paving mixtures by the use of the Nuclear Asphalt Content Gauge.
- 1.2 The paving mixture is examined with a device that utilizes neutron thermalization techniques.
- 1.3 This method may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. **APPARATUS.**

- 2.1 **Asphalt Content Gauge.** Consists of an encapsulated and sealed neutron source such as americium/beryllium, thermalized neutron detectors such as helium-3 or boron tri-fluoride, read-out instrument such as a scaler or a direct-reading digital device calibrated in percent asphalt cement. Variability of the apparatus as 6 percent asphalt cement content shall be no greater than 0.05% for a 4 minute count. The variability of the apparatus is determined from the slope of the calibration curve and the standard deviation of the count rate. Variability is calculated as follows:

$$V = \frac{S.D.}{S}$$

Where:

- V = Apparatus variability, in % asphalt cement  
S.D. = Standard deviation, in counts per automatically timed period  
S = Slope, in counts per % asphalt cement

The standard deviation is calculated from 20 individual automatically timed readings taken on a sample with asphalt cement content with  $\pm 0.5\%$  of the mix design. The range of control mix should be between 2 and 14% asphalt cement by weight.

**NOTE:** The equipment shall be so constructed as to be licensable in accordance with applicable health and safety regulations. Equipment operators shall wear an approved form of radiation dosimetry film badge capable of monitoring the occupational radiation exposure.

**NOTE:** This procedure is sensitive to changes in moisture content. Do not place the equipment where large amounts of hydrogenous material may be moved during calibration or testing procedures (for example, water or plastic materials).

**NOTE:** Keep any other source of neutron radiation at least 30 feet (10 m) from the equipment.

**NOTE:** The asphalt content gauge is calibrated to its surrounding environment through the background count. Take new background counts at the frequencies specified in section 3 and section 6 of this procedure.

**NOTE:** Nuclear devices are subject to long term aging of the radioactive source, detectors, and other electronic systems, which may change the relationship between count rate and asphalt cement content. Because of this aging, new calibration curves should be run each year.

- 2.2 **Oven.** Capable of maintaining the temperature at 221 to 347°F (105 to 175°C).
- 2.3 **Balance.** Minimum capacity of 10,000 grams with sensitivity of 0.1 gram and accuracy of 0.2 gram.
- 2.4 **Straightedge.** Steel, approximately 18 inches (460 mm) in length.
- 2.5 **Plywood.** 3/4 inch (19 mm) or heavier, or metal plate, 3/8 inch (9.5 mm) or heavier having an area slightly larger than the top of the sample pans.
- 2.6 **Sample Pans.** Designed for use with asphalt content gauge.

### 3. CALIBRATION.

- 3.1 This method is sensitive to the type of aggregate, the percentage and source of asphalt cement, and the aggregate gradation. Accordingly, a calibration curve must be developed for each mix design. The curve can be established with four or more points.
- 3.2 Sample the aggregates in accordance with AASHTO T 2. Enough aggregate should be obtained for a minimum of six (6) samples. Dry aggregates to a constant mass in an oven at a temperature of 221 to 347°F (105 to 175°C).
- 3.3 Blend the aggregates in the proper proportions and place the aggregate and asphalt cement in an oven at a temperature of 324°F (162 ± 5°C) for a minimum of one hour.
- 3.4 Place the dry hot aggregate in a tared sample pan in three layers. For each layer, raise and drop the pan approximately one inch, four times. Be sure that the pan bottom strikes evenly. Use a spatula to distribute the aggregate to avoid segregation. Fill the pan with the third layer slightly above the top rim. Place the straightedge firmly across the rim, and using a sawing motion, strike off the surface of the sample so that it is flush with the rim. Gaps between the straightedge and the sample shall be filled with fine aggregate and the sample leveled. Record the mass of the sample as the "Blank Weight." This is the mass that is to be used for all calibration and test samples.
- 3.5 Place the blank sample in the gauge and follow the manufacturer's recommendations to obtain sample counts. Counts for blank samples shall be taken for a minimum of 16 minutes.
- 3.6 All bowls, sample pans, and tools should be heated to approximately 324°F (162°C). Mix a minimum of five (5) asphalt concrete samples. Mix one at the design asphalt cement content, one 0.6% above, one 0.6% below, one 1.2% above, and one 1.2% below. Use the same grade and type of asphalt cement and additives as will be used in the asphalt concrete mixture to be tested.

- 3.7 Perform a background count immediately prior to starting a new calibration. Store and record this count. This background count will be used for the duration of this calibration and become an integral part of the calibration. Counts for background shall be taken for a minimum of 16 minutes.
- 3.8 Begin the calibration using the asphalt concrete mixture sample with the lowest asphalt cement content. Fill the sample pan in three (3) equal layers evenly distributing the sample in the pan. For each layer, level the asphalt concrete mixture with a trowel or spatula to ensure uniform compaction. Fill the pan until the asphalt concrete mixture equals the "Blank Weight." Use the metal plate or plywood to consolidate the asphalt concrete mixture until it is even with the top edge of the pan. This should be done by placing the pan on the floor, placing the plate on top of the sample pan and standing on the plate. Waxed paper may be placed between plate and sample to prevent sticking. All specimens should be compacted at nearly the same temperature to ensure that the mix will compact properly.
- 3.9 Measure and record the temperature of the sample. This is the temperature ( $\pm 9^{\circ}\text{F}$  ( $\pm 5^{\circ}\text{C}$ )) at which all calibration samples and mix samples must be counted.
- 3.10 Place the sample pan containing the asphalt concrete mixture sample in the gauge and proceed as per manufacturer's instructions for operation of the equipment and sequence of operation. Counts for calibration samples shall be taken for a minimum of 16 minutes. Repeat for the remainder of the calibration samples.
- 3.11 Prepare a calibration curve by plotting the calibration asphalt concrete mixture sample counts versus asphalt cement content on linear graph paper, choosing convenient scale factors for counts and asphalt cement content. Some gauges will establish the calibration curve internally by calculating constants which identify the curve, making it unnecessary to plot the curve on graph paper. To be considered acceptable, a calibration should have a correlation factor greater than or equal to 0.995.

$$\text{Correlation Factor} = \frac{n (\sum xy) - (\sum x) (\sum y)}{[n (\sum x^2) - (\sum x)^2] [n (\sum y^2) - (\sum y)^2]}$$

Where:

- n = Number of calibration samples
- x = Percent asphalt cement
- y = Log of the sample count

- 3.12 A separate calibration is required for each asphalt concrete mix design.  
  
**NOTE:** This procedure can be used with mix designs containing Recycled Asphalt Pavement (RAP). To compensate for the binder in the RAP the binder content of the RAP must first be determined by Method C or Method D of this test procedure. This determination is normally performed by the laboratory initially performing the mix design.
- 3.13 A calibration curve may be transferred from one gauge to another provided the two gauges have been properly cross calibrated using the gauge manufacturer's instructions.

#### 4. PREPARATION OF SAMPLE.

- 4.1 The bituminous mixture used for preparing the test sample will be obtained in accordance with AASHTO T 168. The test sample will be prepared by splitting or quartering this material, in accordance with AASHTO T 248, to the appropriate size as determined in section 3.4.



- 4.2 The sample to be tested shall be the same weight as the calibration samples and shall be placed in the sample pans by the same manner used for the calibration samples.
- 4.3 If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in a large flat pan and warm to  $257 \pm 9^{\circ}\text{F}$  ( $125 \pm 5^{\circ}\text{C}$ ) for 25 minutes. The sample shall not be heated for more than 1 hour.

5. **MOISTURE AND VOLATILES DETERMINATION.**

- 5.1 Split or quarter the remaining mixture to obtain a test sample for moisture and volatile determination. The size of the test sample should be  $1,000 \pm 250$  grams. Place the test sample in a tared pan and determine the mass to the nearest 0.1 gram and record as  $W_{1m}$ . Dry to a constant mass in an oven at a temperature of  $257 \pm 9^{\circ}\text{F}$  ( $125 \pm 5^{\circ}\text{C}$ ). Determine the mass of the sample to the nearest 0.1 gram and record as  $W_{5m}$ . The moisture and volatiles weights are used in the determination of bitumen content in section 7.

**NOTE:** The same test specimen may be used for the moisture and volatiles determination and bitumen determination.

6. **PROCEDURE.**

- 6.1 Turn on the equipment and allow for stabilization of the equipment in accordance with the manufacturer's recommendations.
- 6.2 Perform a new background count. A new background count shall be performed at least once a day or whenever the gauge is moved or the environment surrounding the gauge changes. Counts for background shall be taken for a minimum of 16 minutes.
- 6.3 Using a hot asphalt concrete mixture sample, fill the sample pan in three equal layers, evenly distributing the sample in the pan. For each layer, level the asphalt concrete mixture with a trowel or spatula to ensure uniform compaction. Fill the pan until the weight of the asphalt concrete mixture in the pan equals the "Blank Weight." Use the metal plate or plywood to compact the asphalt concrete mixture until it is even with the top of the pan. This should be done by placing the pan on a clean floor, placing the plate on top of the sample pan and standing on the plate.
- 6.4 Place the sample in the gauge and follow the manufacturer's instructions to obtain sample counts and apparent asphalt cement content. Counts shall be taken for a minimum of 8 minutes.
- 6.5 A dry aggregate count should be made often enough to ensure that changes in aggregate do not occur unnoticed. Sample and prepare the aggregates for the dry aggregate count as in sections 3.2 , 3.3 and 3.4. Perform the dry aggregate count as in section 3.5. If a significant change is noted in this count ( $\pm 0.5\%$ ), a new calibration should be run.

7. **CALCULATIONS.**

$$\% AC = \frac{\frac{(P_{5m} \times W_{1m})}{100} - (W_{1m} - W_{5m})}{W_{5m}} \times 100$$

Where:

$P_{5m}$  = Apparent asphalt content from gauge  
 $W_{1m}$  = Mass of moisture sample before drying  
 $W_{5m}$  = Mass of moisture sample after drying

**NOTE:** If the same test specimen is used for bitumen content and moisture and volatiles determination, then:

$$\% AC = P_{5m}$$

**METHOD C**

1. **SCOPE.**

- 1.1 This method covers the determination of bitumen content in bituminous paving mixtures and pavement samples by centrifuge extraction. Trichloroethylene or 1,1,1-trichloroethane solvents are used to extract the asphalt binder from the aggregate. The bitumen content is expressed as a percent of the moisture-free mixture. Aggregate obtained by these methods may be used for sieve analysis using AASHTO T 30.
- 1.2 This method may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety

2. **APPARATUS.**

- 2.1 **Oven.** Capable of maintaining a temperature of  $257 \pm 9^{\circ}\text{F}$  ( $125 \pm 5^{\circ}\text{C}$ ).
- 2.2 **Pan.** Flat bottom, appropriate size.
- 2.3 **Balances.** Minimum capacity of 4,000 grams with sensitivity of 0.1 gram and accuracy of 0.2 gram. Minimum capacity of 250 grams with sensitivity of 0.01 gram and accuracy of 0.02 gram.
- 2.4 **Hot Plate.** Electric, with adjustable heating range.
- 2.5 **Beaker.** Graduated, appropriate size.
- 2.6 **Cylinder.** Graduated, appropriate size.

- 2.7 **Extraction Apparatus.** Consisting of a bowl, approximately that shown in Figure 1 of AASHTO T 164 and an apparatus in which the bowl may be revolved at controlled variable speeds not to exceed 3600 rpm. The apparatus will be provided with a container for catching the solvent thrown from the bowl and a drain for removing the solvent. The apparatus preferably will be provided with explosion-proof features and installed in a hood to provide ventilation.

**NOTE:** Similar apparatus of larger size may be used.

- 2.8 **Filter Rings.** To fit the rim of the bowl. The filter rings will be made from low ash filter paper stock 0.05 inch ( $1.3 \pm .1$  mm) thick. The ash content of the paper should not exceed 0.2%.

### 3. **REAGENTS.**

- 3.1 Trichloroethylene, reagent grade or technical grade Type 1, Federal Specification 0-T-634, latest revision.
- 3.2 1,1,1-Trichloroethane, conforming to Federal Specification 0-T-620a (Int. Amd. 3)

### 4. **PRECAUTIONS.**

- 4.1 The solvents listed in Section 3 should be used only under a hood or with an effective surface exhaust system in a well-ventilated area, since they are all toxic to some degree. Table 1 of AASHTO T 164 lists acceptable OSHA air contaminant limits for these solvents. Materials Safety Data Sheets(MSDS) should be provided by the supplier of the solvents when they are purchased. These sheets should be made readily available to all employees using these solvents. The safety precautions listed in these MSDS sheets should be adhered to at all times.
- 4.2 Trichloroethylene and 1,1,1-trichloroethane in the presence of heat and moisture may form acids that are extremely corrosive to certain metals, particularly when subject to contact over lengthy periods of time. Proper precautions should be taken to not allow these two solvents to remain in small quantities in the effluent tanks of aluminum vacuum extractors.
- 4.3 Trichloroethylene when stored in a steel container and in continuous contact with moisture may decompose by dehydrohalogenation to form unsaturated hydrocarbon liquids and hydrogen chloride. Steel drums containing trichloroethylene should be stored in a cool, dry location, kept tightly sealed and opened as infrequently as possible. The hydrogen chloride in decomposed trichloroethylene may harden an asphalt during the extraction and Absorb recovery test.

### 5. **PREPARATION OF SAMPLE.**

- 5.1 The bituminous mixture used for preparing the test sample will be obtained in accordance with AASHTO T 168. The test sample will be prepared by splitting or quartering this material, in accordance with AASHTO T 248. to the appropriate size as shown in section 5.3.
- 5.2 If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in a large flat pan and warm to  $257 \pm 9^{\circ}\text{F}$  ( $125 \pm 5^{\circ}\text{C}$ ) for 25 minutes. The sample shall not be heated for more than 1 hour.

- 5.3 The recommended size of the test sample is governed by the maximum aggregate size in the mixture and can be found in the table below:

| Maximum Aggregate Size (inches) | Maximum Aggregate Size (mm) | Recommended Mass of Sample (grams) |
|---------------------------------|-----------------------------|------------------------------------|
| 2.5                             | 63                          | 4800 ± 1200                        |
| 2                               | 50                          | 3600 ± 900                         |
| 1.5                             | 37.5                        | 2400 ± 600                         |
| 1                               | 25.0                        | 1800 ± 500                         |
| 3/4                             | 19.0                        | 1400 ± 400                         |
| 1/2                             | 2.5                         | 1000 ± 300                         |
| 3/8                             | 9.5                         | 700 ± 200                          |
| 3/16                            | 4.75                        | 500 ± 200                          |

When the mass of the test sample exceeds the capacity of the equipment used, the test sample may be divided into suitable increments for testing. The results are appropriately combined for calculation of bitumen content.

## 6. MOISTURE AND VOLATILES DETERMINATION.

- 6.1 Split or quarter the remaining mixture to obtain a test sample for moisture and volatile determination. The size of the test sample should be the same as recommended in Section 5.3 or 1,000 ± 250 grams. Place the test sample in a tared pan and determine the mass to the nearest 0.1 gram and record as  $W_{1m}$ . Dry to a constant mass in an oven at a temperature of 257 ± 9°F (125 ± 5°C). Determine the mass of the sample to the nearest 0.1 gram and record as  $W_{5m}$ . The moisture and volatiles weights are used in the determination of bitumen content in section 8.

**NOTE:** Unless recovery of the bitumen from the solution is required, the same test specimen may be used for the moisture and volatiles determination and bitumen determination.

## 7. PROCEDURE.

- 7.1 Place the test sample into the extraction bowl.
- 7.2 Cover the test sample in the bowl with trichloroethylene or 1,1,1-trichloroethane and allow sufficient time for the solvent to disintegrate the sample (not over 1 hour). A small amount of methanol may be added if necessary, to aid extraction. Place the bowl containing the sample and the solvent in the extraction apparatus. Fit the filter ring around the edge of the bowl. Clamp the cover on the bowl tightly and place a beaker under the drain to collect the extract.
- 7.3 Start the centrifuge revolving slowly and gradually increase the speed to a maximum of 3600 rpm or until solvent ceases to flow from the drain. Allow the machine to stop, add 6.76 fl.oz. (200 milliliters) or more of the 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 extract in a suitable manner.

7.4 Carefully remove the contents of the bowl and place the aggregate and the filter ring in a metal pan and dry on a hot plate. When the filter paper has dried, carefully fold and stand it on the aggregate. Burn the filter ring by igniting with a Bunsen burner or match. Continue to dry the aggregate to a constant mass on a hot plate or in an oven at a temperature of  $257 \pm 9^\circ\text{F}$  ( $125 \pm 5^\circ\text{C}$ ).

7.5 **Ash Determination.**

7.5.1 Ashing Method.

7.5.1.1 Determine the volume of the total extract. Agitate the extract thoroughly and immediately measure approximately 3.4 fl.oz. (100 milliliters) or 1/10 of the extract into an ignition dish. Evaporate to dryness on a hot plate. Ash residue at a dull red heat  $932$  to  $1112^\circ\text{F}$  ( $500$  to  $600^\circ\text{C}$ ), cool, and determine the mass of ash in the aliquot (G) to the nearest 0.01 gram.

7.5.2 Centrifuge Method.

7.5.2.1 For this method use any suitable high speed (3000 grams or higher) centrifuge of the continuous-flow type. Determine the mass of a clean empty centrifuge cup or bowl to the nearest 0.01 gram and place in the centrifuge. Position a container at the appropriate spout to catch the effluent from the centrifuging operation. Transfer all of the extract from to an appropriate container suitably equipped with a feed control. 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 9,000 rpm for the SMM type and 20,000+ rpm for the Sharples type). Open the feed line and feed the extract into the centrifuge at a rate of 3.4 to 5.1 fl.oz. (100 to 150 milliliters)/minute. After all the extract has passed through the centrifuge, wash the feed mechanism (with the centrifuge still running) with several increments of clean solvent, allowing each increment to run through the centrifuge until the effluent is essentially colorless. 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 funnel or steam hood and then dry the container in an oven at a temperature of  $257 \pm 9^\circ\text{F}$  ( $125 \pm 5^\circ\text{C}$ ). Cool the container and determine the mass to the nearest 0.01 gram. The increase in mass is the mass of ash in the extract,  $W_4$ .

**NOTE:** The Sharples Supercentrifuge and the SMM continuous flow centrifuge have been found suitable for this method.

**CALCULATIONS.**

7.6 Calculate the mass of ash in the total volume of extract as follows:

$$\text{Total Ash, } W_4 = G \times \frac{V_1}{V_1 - V_2} \quad \text{or} \quad G \times \frac{V_1}{V_3}$$

Where:

- G = Mass of ash in aliquot in grams
- V<sub>1</sub> = Total volume in milliliters
- V<sub>2</sub> = Volume after removing aliquot in milliliters
- V<sub>3</sub> = Volume of aliquot

**NOTE:** When 1/10 of the extract is used, the formula becomes:

$$\text{Total Ash, } W_4 = G \times 10$$

7.7 Calculate the percentage of bitumen in the sample as follows:

$$\% \text{ Bitumen Content of Dry Sample} = \frac{(W_1 - W_2) - (W_3 + W_4)}{W_1 - W_2} \times 100$$

Where:

- W<sub>1</sub> = Mass of original sample
- W<sub>2</sub> = Mass of moisture and volatiles in sample
- W<sub>3</sub> = Mass of extracted mineral matter
- W<sub>4</sub> = Mass of ash in extract

7.8 The moisture and volatiles in the sample are calculated as follows:

$$W_2 = W_1 - \frac{W_{5m} \times W_1}{W_{1m}}$$

Where:

- W<sub>1m</sub> = Mass of moisture sample before drying
- W<sub>5m</sub> = Mass of moisture sample after drying

**NOTE:** When the same sample is used for moisture and volatiles determination and bitumen determination, W<sub>1</sub> = W<sub>1m</sub>

**METHOD D**

**1. SCOPE.**

1.1 This method covers the determination of bitumen content in bituminous paving mixtures and pavement samples by 'reflux' extraction. Trichloroethylene or 1,1,1-trichloroethane solvents are used to extract the asphalt binder from the aggregate. The bitumen content is expressed as a percent of the moisture-free mixture. Aggregate obtained by these methods may be used for sieve analysis using AASHTO T 30.

1.2 This method may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety

**2. APPARATUS.**

2.1 **Oven.** Capable of maintaining a temperature of  $257 \pm 9^{\circ}\text{F}$  ( $125 \pm 5^{\circ}\text{C}$ ).

2.2 **Pan.** Flat bottom, appropriate size.

2.3 **Balances.** Minimum capacity of 4,000 grams with sensitivity of 0.1 gram and accuracy of 0.2 gram.

2.4 **Hot Plate.** Electric, with adjustable heating range.

2.5 **Beaker.** Graduated, appropriate size.

2.6 **Cylinder.** Graduated, appropriate size.

2.7 **Extraction Apparatus.** As shown in Figure 2 of AASHTO T-164.

2.8 **Glass Jar.** Cylindrical, plain, 6 inches (152.4 mm) outer diameter, 18 inches (457.2 mm) high, made of heat-resistant glass.

2.9 **Cylindrical Metal Frames.** Two, 5 inches (127.0 mm) outer diameter and 6.75 inches (171.5 mm) high. The lower frame shall have legs 1.87 inches (47.6 mm) high to support the frame above recesses provided in the top rim of the lower frame. Both frames shall contain 10 to 12 mesh cones with a base of 4.5 inches (114.3 mm) in diameter and 6.75 inches (171.5 mm) side length, mounted inside the top rim of each frame. A bail handle shall be provided on the inside of the top rim of each frame.

2.10 **Condenser.** 6.25 inches (158.8 mm) diameter with 1.25 inches (31.8 mm) inner diameter tubing water inlet and outlet assembled so that no soldered joint comes in direct contact with the condensing solvent vapor.

**NOTE:** Similar apparatus of larger size may be used.

2.11 **Filter Paper,** medium grade, fast filtering, 13 inches (33 cm) in diameter.

**3. REAGENTS.**

3.1 Trichloroethylene, reagent grade or technical grade Type 1, Federal Specification 0-T-634, latest revision.

3.2 1,1,1-Trichloroethane, conforming to Federal Specification 0-T-620a (Int. Amd. 3)

**4. PRECAUTIONS.**

- 4.1 The solvents listed in Section 3 should be used only under a hood or with an effective surface exhaust system in a well-ventilated area, since they are all toxic to some degree. Table 1 of AASHTO T 164 lists acceptable OSHA air contaminant limits for these solvents. Materials Safety Data Sheets(MSDS) should be provided by the supplier of the solvents when they are purchased. These sheets should be made readily available to all employees using these solvents. The safety precautions listed in these MSDS sheets should be adhered to at all times.
- 4.2 Trichloroethylene and 1,1,1-trichloroethane in the presence of heat and moisture may form acids that are extremely corrosive to certain metals, particularly when subject to contact over lengthy periods of time. Proper precautions should be taken to not allow these two solvents to remain in small quantities in the effluent tanks of aluminum vacuum extractors.
- 4.3 Trichloroethylene when stored in a steel container and in continuous contact with moisture may decompose by dehydrohalogenation to form unsaturated hydrocarbon liquids and hydrogen chloride. Steel drums containing trichloroethylene should be stored in a cool, dry location, kept tightly sealed and opened as infrequently as possible. The hydrogen chloride in decomposed trichloroethylene may harden an asphalt during the extraction and Abson recovery test.

**5. PREPARATION OF SAMPLE.**

- 5.1 The bituminous mixture used for preparing the test sample will be obtained in accordance with AASHTO T 168. The test sample will be prepared by splitting or quartering this material, in accordance with AASHTO T 248. to the appropriate size as shown in section 5.3.
- 5.2 If the mixture is not sufficiently soft to separate with a spatula or trowel, place it in a large flat pan and warm to  $257 \pm 9^{\circ}\text{F}$  ( $125 \pm 5^{\circ}\text{C}$ ) for 25 minutes. The sample shall not be heated for more than 1 hour.
- 5.3 The recommended size of the test sample is governed by the maximum aggregate size in the mixture and can be found in the table below:

| Maximum Aggregate Size (inches) | Maximum Aggregate Size (mm) | Recommended Mass of Sample (grams) |
|---------------------------------|-----------------------------|------------------------------------|
| 2.5                             | 63                          | 4800 ± 1200                        |
| 2                               | 50                          | 3600 ± 900                         |
| 1.5                             | 37.5                        | 2400 ± 600                         |
| 1                               | 25.0                        | 1800 ± 500                         |
| 3/4                             | 19.0                        | 1400 ± 400                         |
| 1/2                             | 2.5                         | 1000 ± 300                         |
| 3/8                             | 9.5                         | 700 ± 200                          |
| 3/16                            | 4.75                        | 500 ± 200                          |

When the mass of the test sample exceeds the capacity of the equipment used, the test sample may be divided into suitable increments for testing. The results are appropriately combined for calculation of bitumen content.



6. **MOISTURE AND VOLATILES DETERMINATION.**

- 6.1 Split or quarter the remaining mixture to obtain a test sample for moisture and volatile determination. The size of the test sample should be the same as recommended in Section 5.3 or 1,000 ± 250 grams. Place the test sample in a tared pan and determine the mass to the nearest 0.1 gram and record as  $W_{1m}$ . Dry to a constant mass in an oven at a temperature of 257 ± 9°F (125 ± 5°C). Determine the mass of the sample to the nearest 0.1 gram and record as  $W_{5m}$ . The moisture and volatiles weights are used in the determination of bitumen content in section 7.

**NOTE:** Unless recovery of the bitumen from the solution is required, the same test specimen may be used for the moisture and volatiles determination and bitumen determination.

7. **PROCEDURE.**

- 7.1 Dry in an oven at a temperature of 257 ± 9°F (125 ± 5°C) and determine the mass to the nearest 0.1 gram of two sheets of filter paper with a sample pan. Fold each sheet separately on its diameter and fold twice again, one fold being made over the other to make three segments. Open to form a hollow three-ply cone with a single one-ply seam and place in wire cones of extractor frames.

- 7.2 Place approximately ½ of the sample in each of the filter paper-lined cones.

- 7.3 Pour approximately 20 fl.oz. (600 milliliters) of trichloroethylene into the glass jar and place the frame with the supporting legs in the jar. The solvent level must be below the tip of the cone in this frame. Place the top frame on the bottom frame so that the stub legs fit into the round holes in the top rim of the lower frame.

- 7.4 Place the loaded jar on an electric hot plate and cover the jar with the condenser. Circulate a gentle steady flow of cold water through the condenser. Adjust the heat so that the solvent boils gently and a steady flow of condensed solvent drips into the top cone. Take care to adjust the heat so that the filter cones do not overflow. Continue extraction until the solvent running from the tip of the lower cone appears a light straw color when viewed against a white background. Shut off the heat but not the condenser water, and allow to stand until cool enough to handle.

**NOTE:** If the last few drops of extract are dark, the sample still contains asphalt. This method generally takes 6 to 8 hours to complete.

- 7.5 Remove frames with filter paper and extracted aggregate from jar. Dry the aggregate and filter papers on a hot plate and then to constant mass in an oven at a temperature of 257 ± 9°F (125 ± 5°C) Transfer the extract to a suitable container. Rinse the jar with solvent until clean and add the solvent to the extract.

- 7.6 Ash Determination.

- 7.6.1 Ashing Method.

- 7.6.1.1 Determine the volume of the total extract. Agitate the extract thoroughly and immediately measure approximately 3.4 fl.oz. (100 milliliters) or 1/10 of the extract into an ignition dish. Evaporate to dryness on a hot plate. Ash residue at a dull red heat 932 to 1112°F (500 to 600°C), cool, and determine the mass of ash in the aliquot (G) to the nearest 0.01 gram.

7.6.2 Centrifuge Method.

7.6.2.1 For this method use any suitable high speed (3000 grams or higher) centrifuge of the continuous-flow type. Determine the mass of a clean empty centrifuge cup or bowl to the nearest 0.01 gram and place in the centrifuge. Position a container at the appropriate spout to catch the effluent from the centrifuging operation. Transfer all of the extract from to an appropriate container suitably equipped with a feed control. 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 9,000 rpm for the SMM type and 20,000+ rpm for the Sharples type). Open the feed line and feed the extract into the centrifuge at a rate of 3.4 to 5 fl.oz.(100 to 150 milliliters)/minute. After all the extract has passed through the centrifuge, wash the feed mechanism (with the centrifuge still running) with several increments of clean solvent, allowing each increment to run through the centrifuge until the effluent is essentially colorless. 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 funnel or steam hood and then dry the container in an oven at a temperature of  $257 \pm 9^{\circ}\text{F}$  ( $125 \pm 5^{\circ}\text{C}$ ). Cool the container and determine the mass to the nearest 0.01 gram. The increase in mass is the mass of ash in the extract,  $W_4$ .

**NOTE:** The Sharples Supercentrifuge and the SMM continuous flow centrifuge have been found suitable for this method.

8. **CALCULATIONS.**

8.1 Calculate the mass of ash in the total volume of extract as follows:

$$\text{Total Ash, } W_4 = G \times \frac{V_1}{V_1 - V_2} \quad \text{or} \quad G \times \frac{V_1}{V_3}$$

Where:

- G = Mass of ash in aliquot in grams
- $V_1$  = Total volume in milliliters
- $V_2$  = Volume after removing aliquot in milliliters
- $V_3$  = Volume of aliquot

**NOTE:** When 1/10 of the extract is used, the formula becomes:

$$\text{Total Ash, } W_4 = G \times 10$$

8.2 Calculate the percentage of bitumen in the sample as follows:

$$\% \text{ Bitumen Content of Dry Sample} = \frac{(W_1 - W_2) - (W_3 + W_4)}{W_1 - W_2} \times 100$$

Where:

$W_1$  = Mass of original sample

$W_2$  = Mass of moisture and volatiles in sample

$W_3$  = Mass of extracted mineral matter

$W_4$  = Mass of ash in extract

The moisture and volatiles in the sample are calculated as follows:

$$W_2 = W_1 - \frac{W_{5m} \times W_1}{W_{1m}}$$

Where:

$W_{1m}$  = Mass of moisture sample before drying

$W_{5m}$  = Mass of moisture sample after drying

**NOTE:** When the same sample is used for moisture and volatiles determination and bitumen determination,  $W_1 = W_{1m}$