

Method of Test for  
**BITUMEN CONTENT OF PAVING MIXTURES BY  
REFLUX EXTRACTOR**  
DOTD Designation: TR 307M/307-97

I. **Scope**

- A. This method of test is designed to determine, by hot solvent extraction, the percentage of bitumen in a paving mixture. The mineral matter recovered from this test can be used for sieve analysis. DOTD TR 323 may be used as an alternate to this method.

*Note 1: Although "bitumen" by definition is material soluble in carbon disulfide, 1,1,1, trichloroethane is recommended for use in this method for safety reasons. It normally produces the same results within the precision of the method. Other solvents, or solvent mixtures containing alcohol, may be substituted in this method and similar results may be obtained, but the relationship of such results to those obtained with 1,1,1 trichloroethane or carbon disulfide cannot be predicted or assumed. The addition of 10 percent denatured ethyl alcohol to 1,1,1 trichloroethane facilitates the extraction of asphalt from the asphalt paving mixtures.*

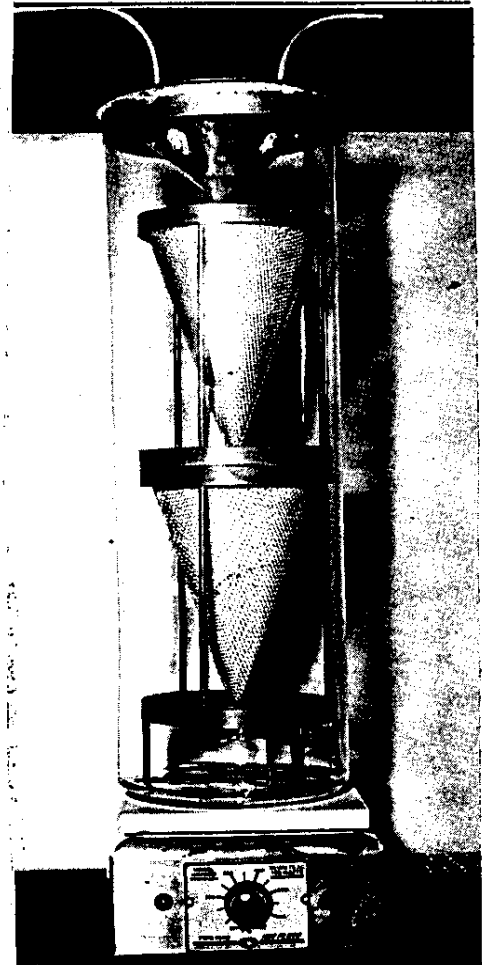
B. **Reference Documents**

1. DOTD TR 108, Splitting and Quartering Samples.
2. DOTD TR 314, Ash Content of Asphaltic Concrete Mixture Solvent Obtained by Reflux or Centrifuge Extraction.
3. DOTD TR 323, Determining the Asphalt Content of Asphaltic Mixtures by the Ignition Method
4. AASHTO T 164, Quantitative Extraction of Bitumen from Bituminous Paving Mixtures.

II. **Apparatus**

- A. **Extraction apparatus - Alternate No. 1 (Figure 1)**
1. **Glass jar** - cylindrical, plain 150 mm (6 in.) outside diameter and 450 mm (18 in.) high made of Pyrex or

2. **other heat resistant glass.**
2. **Electric hot plate** - thermostatically controlled, with at least a 1500 mm (6 in.) square heating surface and 700 watts capacity, or a rheostat controlled electric heater.



**Figure 1**  
**Reflux Extractor**

3. **Two cylindrical frames** - 130 mm (5 in.) outside diameter and 120 mm (6 3/4 in.) high. The lower frame legs shall be 50 mm (1 7/8 in.) high to support frame above solvent level. The upper frame shall have

stub legs which fit in recesses provided in the top rim of the lower frame. Both frames shall be fabricated with metal cones that will provide suitable support for the filter paper and sample, such as wire mesh, metal rods, etc. Cones shall be of sufficient diameter at the top to fit the frame, have a side length of approximately 170 mm (6 3/4 in.), and be mounted inside the top rim of each frame. A bail handle shall be provided on the top rim of each frame. All metal shall be of a non-corrosive type such as brass, stainless steel, etc.

4. **Condenser** - made from sheet copper of a minimum thickness of 0.8 mm (0.03 in.), to close the mouth of the jar, and equipped with suitable inlet and outlet tubes for the cooling water. The bottom surface of the condenser should be designed to obtain the most effective condensation of the solvent vapor and return of the solvent to the upper frame. If soldered joints on the condensing surface are necessary, the joints should be tight with minimum exposure to solder. Other types of condensers which will effectively return the hot solvent to the mixture may be used.
  5. **Filter paper** - medium grade, fast filtering, 330 to 360 mm diameter.
- B. Extraction apparatus** - Alternate No. 2
1. **Glass jar** - cylindrical, plain 220 mm (8 3/4 in.) outside diameter and 450 mm (18 in.) high of Pyrex or other heat resistant glass.
  2. **Electric hot plate** - thermostatically controlled, with at least a 230 mm (9 in.) square heating surface and 700 watts capacity, or a rheostat controlled electric heater.
  3. **Two cylindrical frames** - 200 mm (7 3/4 in.) outside diameter and 170 mm (6 3/4 in.) high. The lower frame legs shall be 50 mm (1 7/8 in.) high to support frame above solvent level. The upper frame shall have stub legs which fit in recesses provided in the top rim of the lower

frame. Both frames shall be fabricated with metal cones that will provide suitable support for the filter paper and sample, such as wire mesh, metal rods, etc. Cones shall be of sufficient diameter at the top to fit the frame, have a side length of approximately 150 to 170 mm (6 to 6 3/4 in.), and be mounted inside the top rim of each frame. A bail handle shall be provided on the top rim of each frame. All metal shall be of a non-corrosive type such as brass, stainless steel, etc.

4. **Condenser** - same as in step II.A.4 above, except size, which shall be sufficiently large enough to close the mouth of the glass jar.
  5. **Filter paper** - medium grade, fast filtering, 330 to 360 mm diameter
  6. **Oven** - capable of maintaining a temperature of  $135 \pm 5^{\circ}\text{C}$  ( $275 \pm 9^{\circ}\text{F}$ )
  7. **Hot plate** - The use of hot plate drying in the field is allowed where ovens are not available or practical. Open flame hot plates shall be equipped with a suitable shield to evenly disperse the heat and avoid direct contact of the flame with the drying pan.
  8. **Balance** - capable of reading to 2000 g and sensitive to 0.1 g.
- C. **Reagents** - 1,1,1 trichloroethane
- D. **Personal protective equipment** - gloves, eye protection, etc.
- E. **Worksheet** - Asphaltic Concrete Plant Report, DOTD Form No. 03-22-3085.

### III. Health Precautions

The solvent listed above is to be used under a hood or with an effective surface exhaust system. Material Safety Data Sheets (MSDS) shall be maintained at locations where employees handle hazardous materials. Extreme caution is to be exercised when testing this material due to the high temperature involved.

The solvent used is toxic and should only be used with proper ventilation. In no event shall it be used to clean hands. The solvent shall be stored away from all testing equipment to prevent corrosion damage.

#### IV. Sample

- A. If the sample is not sufficiently soft to separate with a trowel or spatula, heat at a temperature of  $135 \pm 5^\circ \text{C}$  ( $275 \pm 9^\circ \text{F}$ ) until it can be handled and quartered. For mixtures containing liquid asphalts and for cutbacks and emulsified asphalt used in cold mixtures, it will be necessary to remove their fluxing agents to determine the bitumen content. This shall be done by heating the mixture slowly to evaporate the fluxing agents. Take care not to under heat the mixture, thereby, not removing all of the fluxing agent, or overheating it, causing the asphalt to oxidize. In mixtures where a rapid curing cutback asphalt is used, take care not to overheat the mixture causing the inflammable fluxing agent to catch fire and burn.

*Note 2: Exercise extreme caution when using a hot plate to ensure adherence to the above conditions.*

- B. Reduce sample to the appropriate size by quartering in accordance with DOTD TR 108, Method B.
1. Do not use absorbent materials as a quartering surface.
  2. Do not attempt selection of samples of an exact predetermined mass.
- C. Determine the mass of the test samples to the nearest 0.1 g and minimum size of:
1. Gravel, slag, stone, shell. Minimum: 900 g.
  2. Expanded clay. Minimum: 630 g.

#### V. Procedure

- A. Dry and determine the mass of the filter paper(s) to the nearest 0.1 g.
1. When extraction apparatus Alternate No. 1 is used:
    - a. Dry and determine the mass of two sheets of filter paper.
    - b. Fold each separately on its diameter and fold twice again, one fold being made over the other to make three segments. (Figure 2)
    - c. Open to form a hollow 3-ply cone with a single 1-ply seam.
    - d. Place in wire cones of extractor frames.

2. When extraction apparatus Alternate No. 2 is used:
  - a. Dry and determine the mass of a sheet of filter paper as stated above for each cone used.
  - b. Fold on its diameter and fold once again.
  - c. Open to form a hollow cone having a 3-ply side and one 1-ply side.
  - d. Place in metal cone of extractor frame.
- B. Distribute the prepared sample as necessary in the filter paper lined cones and compact by tapping lightly with a finger or spatula blade.
- C. Assemble the loaded frame in the jar. Carefully pour approximately 500 mL solvent over the top frame (use approximately 1500 mL for the large extractor). At no time should the solvent level contact the tip of the cone in the lower frame. Four or five glass beads placed into the jar will facilitate easy boiling of solvent.
- D. Place loaded jar on electric hot plate, cover with condenser and circulate cold water through it, regulating to a gentle steady flow.
- E. Adjust heater so that solvent boils gently and a steady flow of condensed solvent drips into the top cone. Care must be taken to adjust heat so that the material in each cone is covered with solvent and, at the same time, the filter cones do not overflow.



Figure 2  
Folding Filter Paper

- F. Continue extraction until solvent running from tip of lower cone appears colorless when viewed against a white background; then shut off heat, but not water through condenser, and allow to stand until cool enough to handle.
- G. Remove frames from jar, dry first in air and then to constant mass in an oven adjusted to 135±5°C (275±9°F). If drying at this temperature causes the filter papers to become so brittle they cannot be handled and brushed, dry them separately from the aggregate and at a lower temperature.

**Note 3:** Constant mass for drying purposes is defined as less than 0.1% mass loss between successive mass determinations no less than 5 minutes apart.

**Note 4:** The filter paper and aggregate may be carefully separated from the frame and transferred to a pan for drying and/or determining mass.

- H. After drying, the filter papers shall be cleaned by brushing of all adhering aggregate, returning this material to the sample. Determine the mass of the extracted sample and filter papers to the nearest 0.1 g.

**Note 5:** Since the original sample mass was obtained at a temperature near 135°C (275°F), the mass of the extracted sample and filter papers should be determined as soon as possible after removal from the drying oven.

**VI. Calculations**

Calculate the percent asphalt in the sample to the nearest 0.1% using the following formula.

$$\text{Asphalt, \%} = \frac{Q - (R + V + U)}{Q} \times 100$$

where:

- Q = wt of mix (with AC), g
- R = wt of dry agg. (minus AC), g
- V = a correction factor equal to the amount of mineral matter (ash) in the solvent as determined by the laboratory using DOTD TR 314, g (correction factor needs to be determined on at least each JMF.)
- U = gain in filter wt, g
- 100 = constant

example:

- Q = 1006.3
- R = 958.2
- V = 0.2
- U = 1.2

$$\begin{aligned} \text{Asphalt, \%} &= \frac{1006.3 - (958.2 + 0.2 + 1.2)}{1006.3} \times 100 \\ &= \frac{1006.3 - 959.6}{1006.3} \times 100 \\ &= \frac{46.7}{1006.3} \times 100 \\ &= 0.04640 \times 100 \\ &= 4.640 \\ \text{Asphalt, \%} &= 4.6 \end{aligned}$$

**VII. Report**

Report the percentage of bitumen to the nearest 0.1 percent.

**VIII. Normal Test Reporting Time**

The normal test reporting time is 12 hours.