1. **SCOPE**

1.1. **Description of Test**

This method covers the quantitative determination of bitumen in hot-mixed paving mixtures and pavement samples.

1.2. **Application of Test**

This method can be used for quantitative determination of bitumen on hot-mixed paving mixtures and pavement samples for specification acceptance, service evaluation, control and research.

1.3. **Units of Measure**

The bitumen content is expressed as mass percent of moisture-free mixtures.

2. **APPARATUS AND MATERIALS**

2.1. **Equipment Required**

Oven capable of maintaining the temperature of 110 ± 5° C.

Balance, accuracy of 0.1g.

Extraction apparatus, similar to that shown in Figure 1.

Glass jar, cylindrical, plain, made of heat resistant glass.

Cylindrical metal frames, one or two. The lower frame shall have legs of sufficient length to support the frame above the solvent level. The legs of the upper frame shall fit securely in the top rim of the lower frame. The metal used in the frames shall be inactive to the solvents used in the test.

Condenser, hemispherical condensing surface. The material used shall be unreactive to water and to the solvent used and shall be provided with suitable water inlet and outlet.

Filter paper, medium grade, fast-filtering with diameter such that it will completely line the metal cones in the frames.
Electric hot plate, thermostatically controlled, of sufficient dimensions and heat capacity to permit refluxing of the solvent.

FIGURE 1
2.2 Materials Required

1,1,1 – Trichloroethane

2.3 Sample to be Tested

Obtain samples to be tested as per STP 103.

3. PROCEDURE

3.1 Sample Preparation

If the mixture is not soft enough to separate with a spatula or trowel, place it in a large, flat pan and warm to 110° C only until it can be handled or mixed. Split or quarter the material until the mass of material required for the test is obtained. A test specimen for moisture determination should be obtained in this same manner from the remaining sample.

3.2 Test Procedure

Determine the moisture content of the mixture in accordance with the test method STP 204-1.

Dry and determine the mass of one sheet of filter paper for each frame to be used. Fold each paper on its diameter, fold the ends over and spread it open to form a proper size to fit inside the metal cones.

Determine the mass of each frame with its filter paper liner to the nearest 0.5 g. Record the mass, identifying each frame by number.

Place the test portion in the frame. If two frames are used distribute the test portion equally between the two. The top of the test portion must be below the upper edge of the paper liner. Determine the mass of each loaded frame separately to the nearest 0.5 g. Again, record the mass.

Pour the solvent into the glass cylinder and place the bottom frame into it. The solvent level should be below the apex of the one in the lower frame. If two frames are used, place the upper frame in the lower frame, fitting its legs into the holes in the upper rim of the lower frame.

Place the glass cylinder on the hot plate. Cover the condenser. Circulate a gentle steady stream of cool water through the condenser. Adjust the temperature of the hot plate so that the solvent will boil gently and a steady stream of condensed solvent flows into the
If necessary, adjust the temperature of the hot plate to maintain the solvent stream at a rate necessary to keep the test portions in the cones completely covered with condensed solvent. Take care not to allow condensed solvent to overflow the filter cones. Continue the refluxing until the solvent flowing from the lower cones is light straw colour. At this point, turn off the hot plate and allow the apparatus to cool with the water running the condenser. When boiling has ceased and the cylinder is cool enough to handle, turn off the condenser and remove from the cylinder.

Remove the frame assembly from the cylinder. Allow to dry in air and dry to constant mass in an oven at 110°C.

Determine the amount of mineral matter in the extract by any of the following test methods:

Ashing Method – record the volume of the total extract. Determine the mass of an ignition dish. Agitate the extract thoroughly and immediately measure 100 ml into the ignition dish. Evaporate to dryness on a steam bath or hot plate. Ash residue at a dull red heat (500 to 600°C), cool and add 5 ml of saturated ammonium carbonate solution per gram of ash. Digest at room temperature for 1 hour. Dry in an oven at 100°C to constant mass, cool in a desiccator, and determine the mass. Calculate the mass of mineral matter in the total volume of extract, W₄, as follows:

$$W₄ = G \left[ \frac{V₁}{V₁ - V₂} \right]$$

Where:
- \(G\) = ash in aliquot, g
- \(V₁\) = total volume, ml
- \(V₂\) = volume after removing aliquot, ml

Centrifuging Method – determine the mass of a clean empty centrifuge cup to 0.01 g 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 to an appropriate feed container. To ensure quantitative transfer of the extract to the feed container, the receptacle containing the extract should be washed several times. Start the centrifuge and allow to reach a constant operational speed. Open the feed line and feed the extract into the centrifuge at a rate of 100 to 150 ml/minute. After all the extract has passed through the centrifuge, wash the feed mechanism (with centrifuge still running) with several increments of clean solvent. Allow the centrifuge to stop and remove the cup. Clean the outside. Allow the residual solvent to evaporate and then dry the container in an oven controlled at 110°C. Cool the container and redetermine the mass immediately. The increase in mass is the mass of mineral matter, \(W₄\), in the extract.
Volumetric Method – place the extract in a previously tared and calibrated flask. Place the flask in a controlled temperature bath controlled to ± 0.1°C and allow to come to the temperature at which the flask was calibrated. When the desired temperature has been reached, fill the flask with solvent that has been kept at the same temperature. Bring the level of the fluid in the flask up to the neck, insert the stopper, making sure the liquid overflows the capillary, and remove from the bath. Wipe the flask dry, determine the mass to the nearest 0.1 g and record this mass as the mass of flask plus extract. Calculate the volume of asphalt and fines in the extract as follows:

\[
V_1 = V_2 - \left( \frac{M_1 - M_2}{G_1} \right)
\]

Where:
- \( V_1 \) = volume of asphalt and fines in the extract
- \( V_2 \) = volume of the flask
- \( M_1 \) = mass of the contents of the flask
- \( M_2 \) = mass of the asphalt and fines in the extract
  = mass of the total samples minus the mass of the extracted aggregate
- \( G_1 \) = specific gravity of the solvent

Calculate the mass of fines in the extract as follows:

\[
M_3 = K (M_2 - G_3 V_1)
\]

Where:
- \( M_3 \) = mass of fines in the extract
- \( G_3 \) = specific gravity of asphalt
- \( G_2 \) = specific gravity of fines

\[
K = \frac{G_2}{G_2 - G_3}
\]

\[
V_1 = \text{volume of asphalt and fines in the extract}
\]

\[
M_2 = \text{mass of the asphalt and fines in the extract}
\]

\[
= \text{mass of the total samples minus the mass of the extracted aggregate}
\]

4. RESULTS AND CALCULATIONS

4.1 Calculations

Calculate the percent bitumen in the test portion as follows:
Bitumen Content, % = \left( \frac{(W_1 - W_2) - (W_3 - W_4)}{W_1 - W_2} \right) \times 100

Where: 
\begin{align*}
W_1 &= \text{mass of test portion} \\
W_2 &= \text{mass of water in the test portion} \\
W_3 &= \text{mass of the extracted mineral aggregate} \\
W_4 &= \text{mass of the mineral matter in the extract}
\end{align*}

4.2 **Reporting Results**

Report the percent moisture and percent bitumen on appropriate form MR-70 or EPS-90.

5. **CALIBRATIONS, CORRECTIONS, REPEATABILITY**

5.1 The amount of asphalt extracted must be adjusted for the following items:

Extraction error required because of the loss of fines through the filter and for asphalt absorbed into the aggregate.

Moisture correction required because the moisture in the mix is extracted as bituminous binder.

5.2 The single laboratory standard deviation has been found to be 0.18%. Therefore, results of two properly conducted tests by the same operator should not differ by more than 0.52%. The multi-laboratory standard deviation has been found to be 0.29%. Therefore, the results of two properly conducted tests from two different laboratories on samples from the same batch should not differ by more than 0.81%.

5.3 All equipment should be cleaned frequently to prevent testing errors.

6. **ADDED INFORMATION**

6.1 **References**

ASTM D-2172

Alberta Transportation and Utilities Procedure ATT-12/88. Part 11, Filterless extraction and filterless centrifuge method.
6.2 **Safety**

Fumes from trichloroethane are toxic therefore every effort should be made to have good ventilation while the test is being performed.

Respirators and gloves should be worn when handling trichloroethane.

6.3 **WHMIS**

The First Aid and Precautions and Storage guidelines for trichloroethane are outlined in the Chemical Safety Data Sheet. Employees using trichloroethane are responsible for and must be aware of the information contained in the Chemical Safety Data Sheets. A brief summary follows:

**FIRST AID – TRICHLOROETHANE**

**INHALATION:** Remove to fresh air. Give artificial respiration if necessary. Have trained personnel give oxygen if breathing is difficult. Keep warm and at rest. Obtain immediate medical attention.

**INGESTION:** Give 2 or 3 glasses of milk or water, induce vomiting. Keep warm and at rest. Obtain immediate medical attention.

**EYES:** Immediately flush with a gentle stream of water for at least 15 minutes, lifting upper and lower lids occasionally. Obtain medical attention if irritation persists after flushing.

**SKIN:** Wash exposed skin thoroughly with water while removing contaminated clothing. Obtain medical attention if irritation persists.

**REACTIVITY AND STORAGE - TRICHLOROETHANE**

**INCOMPATIBLE WITH:** Water, Oxidizing Material, chemically active metals and aluminum.

**PRECAUTIONS:** Avoid heat and open flames. Store in a cool, well ventilated area. Keep container tightly closed.