

5.16.33 BITUMEN CONTENT OF PAVING MIXTURES BY REFLUX EXTRACTION
(Kansas Test Method KT-33)

a. SCOPE

This method covers the procedure for the quantitative determination of bitumen in bituminous paving mixtures and pavement samples. The asphalt is extracted using 1,1,1-trichloroethane as a solvent. The bitumen content is calculated as the difference between the weight of the extracted aggregate, moisture content, and ash and the weight of the total mix. KT-33 reflects testing procedures found in AASHTO T 164.

b. EQUIPMENT

b.1. Oven capable of maintaining a uniform temperature of approximately $110 \pm 5^{\circ}\text{C}$ ($230 \pm 9^{\circ}\text{F}$).

b.2. Balance, capable of weighing 2 000 g with a readability of 0.1 g and a sensitivity of 0.05 g.

b.3. Hot plate, electric, thermostatically controlled with appropriately sized aluminum cake pan.

b.4. Battery Jar, cylindrical, plain, 150 mm (6 in) OD by 450 mm (18 in) high (for the 1 000 g extraction apparatus) or 220 mm (8 3/4 in) OD by 450 mm (18 in) high (for the 2 500 g extraction apparatus), made of heat-resistant glass.

b.5. Cylindrical Metal Frames for Extraction Apparatus.

b.5.a. 1,000 g Extraction Apparatus: Two, 127 mm (5 in) OD and 171 mm (6 3/4 in) high. The lower frame shall have legs 47.6 mm (1 7/8 in) high to support the frame above the solvent level. The upper frame shall have stub legs which fit in recesses provided in the top rim of the lower frame. The frames for the 1 000 g extraction apparatus shall contain cones of woven wire cloth 2.00 to 1.70 mm (No. 10 to 12) or rods with a base 114 mm (4 1/2 in) in diameter and 170 mm (6 3/4 in) side length, attached inside the top rim of each frame.

b.5.b. 2,500 g Extraction Apparatus: Two, 197 mm (7 3/4 in) OD and 165 mm (6 1/2 in) high. The lower frame shall have legs 57.2 mm (2 1/4 in) high to support the frame above the solvent level. The upper frame shall have stub legs which fit over the top of the lower frame. The frames for the 2

500 g extraction apparatus shall contain cones made of 3 mm (1/8 in) metal rod. The cones shall have a base 190 mm (7 1/2 in) in diameter and a 185 mm (7 1/4 in) side length and are attached inside the top rim of each frame.

b.6. Condensers.

b.6.a. 1,000 g Extraction Apparatus: 159 mm (6 1/4 in) in diameter. The condenser for the 1,000 g extraction apparatus shall have either a hemispherical or a stepped cone type condensing surface equipped with a 6 mm (1/4 in) water inlet and outlet. The ends of the copper tubing shall protrude from the condenser top, serving as a water inlet and outlet.

b.6.a. 2,500 g Extraction Apparatus: 235 mm (9 1/4 in) in diameter. The condenser for the 2,500 g extraction apparatus shall be a conical spiral formed from 3 m (10 ft) of 8 mm (5/16 in) OD copper tubing. The ends of the copper tubing shall protrude from the condenser top, serving as a water inlet and outlet.

b.7. Filter Paper, grade 615, 330 (400) mm in diameter. Grade 617 is acceptable when material possesses a high level of fines.

b.8. A supply of 1,1,1-trichloroethane solvent.

b.9. A supply of Denatured Ethyl Alcohol.

b.10. Appropriate Graduate, 1,000 or 2,000 mL capacity.

b.11. Evaporating Dish, 125 mL capacity.

b.12. Meker Gas Burner capable of maintaining the ash in the ignition dish at a temperature between 500 and 600°C.

b.13. A supply of saturated solution of reagent grade ammonium carbonate, $(\text{NH}_4)_2\text{CO}_3$.

c. SAMPLE PREPARATION

c.1. If necessary, the mixture is placed in a large flat pan and heated in an oven maintained at approximately 110°C (230°F) until it can be readily separated.

c.2. The mixture is then separated by using a trowel or spatula. Care should be taken during the separation process to prevent the fracturing or segregation of the aggregate.

c.3. The bituminous material shall be thoroughly mixed and a sample of approximately 1,000 g (2,500 g for the larger extractor) shall be obtained by a method of quartering, such as the following:

Step 1. Spread a sheet of paper (Kraft or similar) on a hard, clean, smooth and level surface. Place the sample in a pile near the center of the paper and mix by alternately lifting each corner towards the opposite corner thereby rolling the mixture to the opposite corner. This should be performed in a vigorous manner. Placing the sample on a piece of cardboard and mixing thoroughly with a trowel is an acceptable alternate.

Step 2. Divide the pile into 4 equal quarters with a straightedge (trowel or similar metal blade) and completely remove 2 pre-selected diagonally opposite quarters.

Step 3. Continue this quartering procedure until the original sample is reduced to the approximately desired size. On the final quartering step, if the sample is too large before quartering but will be too small after quartering, the sample pile can be divided into different section sizes. In other words, the pile is divided into equal opposite sectors but unequal adjacent sectors. This can be accomplished such that the dividing angle at the center of the sample pile is varied from the normal 90 degrees. Opposite sections can then be selected to obtain the desired sample size.

c.4. During the quartering process, a sample is obtained for moisture determination (KT-16) by the same procedure as indicated in **c.3.** above, except the reduced sample will be approximately 350 g.

d. PROCEDURE

d.1. Fold two sheets of filter paper. Dry to constant weight at approximately 110°C (230°F) and place the filter papers in the cones of the extractor frames. Immediately weigh the frames and filter paper to the nearest 0.1 g.

d.2. Load the sample into the filter paper lined cones, approximately half the sample going into each frame, weigh and record.

d.3. Pour approximately 1,000 mL (2,000 mL) for the larger extractor) of 1,1,1-trichloroethane into the battery jar. Wet each filter paper cone with denatured ethyl alcohol and

immediately place the loaded frames in the battery jar. Place the loaded battery jar on the hot plate and cover the jar with the condenser.

d.4. Circulate a gentle, steady flow of cold water through the condenser. Adjust the hot plate so that the solvent boils gently and a steady flow of condensed solvent drips into the top cone. Care should be taken 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 colorless when viewed against a white background. Shut off the heat but not the condenser water, and allow to stand until cool enough to handle.

d.5. Remove the frames from the battery jar and place the frames, filter papers and the aggregate in a flat pan. Dry to a constant weight in an oven maintained at approximately 110°C (230°F). Weigh to the nearest 0.1 g. If the oven is not vented to the outside, the sample should be air dried outside before placement in the oven. The fumes of 1,1,1-trichloroethane are toxic and care should always be exercised when handling this substance.

d.6. Transfer the extract to a 1,000 or 2,000 mL graduate. Rinse the battery jar with solvent until clean and add to the extract.

d.7. Record the volume of the total extract in the graduate. Agitate the extract thoroughly and immediately remove approximately 100 mL and record the volume. Place the aliquot portion in a previously weighed evaporating dish. Evaporate to dryness in a vented 110°C (230°F) oven. Ash the residue at a dull red heat. This may be accomplished by adjusting the Meker gas burner such that the interior blue flame cone is approximately 60 mm (2 1/2 in) tall and the ignition dish is placed at the top of this flame cone. Cool the residue and add 5 mL of saturated ammonium carbonate, (NH₄)₂CO₃, solution per gram of ash. Allow to stand at room temperature for one hour. Dry in an oven at approximately 110°C (230°F) to a constant weight, cool and weigh.

e. CALCULATION

Calculate the weight of ash in the total extract by the following formula:

$$W_5 = \frac{W_a(V_1)}{(V_2)}$$

Where: W_5 = weight of ash in the total extract.
 W_a = weight of ash in the aliquot portion.
 V_1 = Volume of the total extract.
 V_2 = Volume of the aliquot portion.

Calculate the percentage of bitumen in the sample as follows:

$$\text{Bitumen content of dry sample, percent} = \frac{100(W_1 - W_2 - W_4 - W_5)}{(W_2 - W_3) + W_5}$$

Where: W_1 = Combined weight of filter papers, frames and original sample.
 W_2 = Combined weight of filter papers, frames and aggregate after extraction.
 W_3 = Weight of filter papers and frames.
 W_4 = Weight of moisture in sample (% moisture from KT-16). ($W_4 = \% \text{ moisture} \times [W_1 - W_3]$)
 W_5 = Weight of ash in total extract.