Experiment No. 1
Standard Test Method for
PENETRATION OF BITUMINOUS MATERIALS

Objective: This test method covers determination of the penetration of semi-solid and solid bituminous materials.

Significance and Use: The penetration test is used as a measure of consistency. Higher values of penetration indicate softer consistency.

Relevant Theory: The penetration test gives an empirical measurement of the consistency of a material in terms of the distance a standard needle sinks into that material under a prescribed loading and time. Although more fundamental tests are being substituted for this test, it still may be included in specifications for viscosity of asphalt cements to ensure the exclusion of materials with very low penetration values at 25 °C (77 °F).

Standard Reference: ASTM D5 and AASHTO T49.

Apparatus and Materials Required:

- *Penetration Apparatus* - Any apparatus that permits the needle holder (spindle) to move vertically without measurable friction and is capable of indicating the depth of penetration to the nearest 0.1 mm.
- *Penetration Needle* - The needle shall be made from fully hardened and temper stainless steel, 50 mm in length and 1.00 to 1.02 mm in diameter.
- *Sample container* - A metal or glass cylindrical, flat bottom container of diameter 55 mm and an internal depth of 35 mm.
- *Water bath* - A bath having a capacity of at least 10 liters and capable of maintaining a temperature of 25 ± 0.1 °C.
- *Transfer Dish* - When used, the transfer dish shall have a capacity of at least 350 ml and of sufficient depth of water to cover the large sample container.
- *Thermometers* - Calibrated liquid-in-glass thermometers of suitable range with subdivisions and maximum scale error of 0.1 °C.

Preparation of Test Specimen:

1. Heat the sample with care, stirring when possible to prevent local overheating, until it has become sufficiently fluid to pour. In no case should the temperature be raised to more than 60 °C above the expected softening point.
2. Pour the sample into the sample container to a depth such that, when cooled to the temperature of test, the depth of the sample is at least 10 mm greater than the depth to which the needle is expected to penetrate. Pour two separate portions for each variation in test conditions.
3. Loosely cover each container as a protection against dust and allow cooling in an atmosphere at a temperature between 15 and 30 °C for 1 to 1.5 h.
4. Then place the two samples together with the transfer dish, if used, in the water bath maintained at the prescribed temperature of test for 1 to 1.5 h.

Test Conditions:

Where the conditions of test are not specifically mentioned, the temperature, load, and time are understood to be 25 °C, 100 g, and 5 s, respectively.

Test Procedure:

1. Clean a penetration needle with toluene or other suitable solvent, dry with a clean cloth, and insert the needle in the penetrometer.
2. Unless otherwise specified place the 50-g weight above the needle, making the total moving load 100 ± 0.1 g.
3. Place the sample container in the transfer dish, cover the container completely with water from the constant temperature bath and place the transfer dish on the stand of the penetrometer.
4. Position the needle by slowly lowering it until its tip just makes contact with the surface of the sample. This is accomplished from a properly placed source of light.
5. Either note the reading of the penetrometer dial or bring the pointer to zero.
6. Quickly release the needle holder for the specified period of time and adjust the instrument to measure the distance penetrated in tenths of a millimeter. If the container moves, ignore the result.
7. Make at least three determinations at points on the surface of the sample not less than 10 mm from the side of the container and not less than 10 mm apart.
8. Return the sample and transfer dish to the constant temperature bath between determinations. Use a clean needle for each determination.

Table 1: Precision Criteria

| Material	Standard Deviation or Coefficient of Variation	Acceptable Range of Two Test Results
Single-operator precision: Asphalts at 25 °C below 50 penetration, units	0.35	1
Asphalts at 25 °C, 50 penetration and above, percent of their mean	1.10	3
Multilaboratory precision: Asphalts at 25 °C below 50 penetration, units	1.4	4
Asphalts at 25 °C, 50 penetration and above, percent of their mean	2.8	8

Experimental Data and Results: Tabulate the data in the given *Test Data Sheet.* Report to nearest whole unit the average of three penetrations whose values do not differ by more than the following:

| Penetration	0 to 49	50 to 149
Maximum difference between highest and lowest determination	2	4

Discussion: Discuss about the test in the given *Test Data Sheet.*

Table 2: AASHTO M20 and ASTM D946 Penetration Grades

Penetration Grades	Comments
40 – 50	Hardest Grade
60 – 70	Typical grades used in the U.S
85 – 100	
120 – 150	
200 – 300	Softest grades. Used for cold climates such as northern Canada
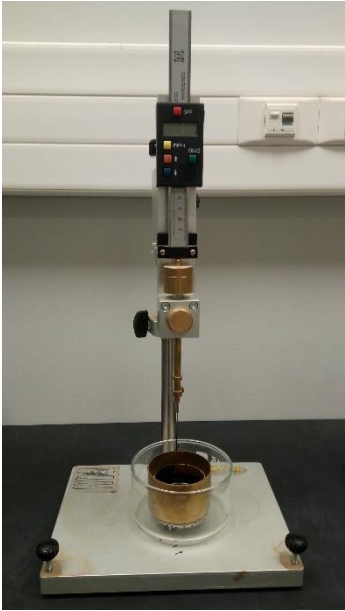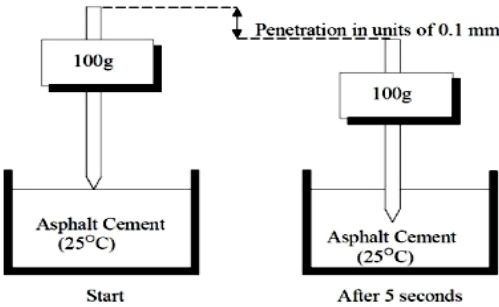
(a) Penetrometer   (b) Needle penetration

Experiment No. 1
Test Data Sheet
PENETRATION OF BITUMINOUS MATERIALS

Sample No. _____   Description of Material: _____

Tested by: _____   Date of Testing: _____

Test Temperature: _____ °C   Total Penetrating Weight on Sample: _____ g.

Container No. _____   Penetrating Time: _____ s

Test No.	Initial Reading	Final Reading	Penetration, units
1			
2			
3			
Average Penetration, units			

Test No.	Initial Reading	Final Reading	Penetration, units
1			
2			
3			
Average Penetration, units			

Test No.	Initial Reading	Final Reading	Penetration, units
1			
2			
3			
Average Penetration, units			

Discussion of Test Results:

_____

_____

_____

_____

_____

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Experiment No. 2
Standard Test Method For
SOFTENING POINT OF BITUMEN

</div>

Objective: This test method covers the determination of softening point of bitumen in range from 30-80 °C using the ring-and-ball apparatus immersed in distilled water.

Significance and Use:

- Bitumens are viscoelastic materials without sharply defined melting points; they gradually become softer and less viscous as the temperature rises. For this reason, softening points must be determined by an arbitrary and closely defined method if results are to be reproducible.
- The softening point is useful in the classification of bitumens, as one element in establishing the uniformity of shipments or sources of supply, and is indicative of the tendency of the material to flow at elevated temperatures encountered in service.

Relevant Theory: The ring-and-ball softening point test is used to measure the susceptibility of blown asphalt to temperature changes by determining the temperature at which the material will be adequately softened to allow a standard ball to sink through it. The test is conducted by first placing a sample of the material to be tested in the brass ring which is cooled and immersed in the water or glycerin bath that is maintained at a temperature of 5°C (41°F).

Standard Reference: ASTM D 36 and AASHTO T 53

Apparatus and Material Required:

- *Rings* - Two square-shouldered brass rings conforming to the standard dimensions
- *Pouring Plate* - A flat, smooth, brass plat approximately 50 by 75 mm (2 by 3 in.)
- *Balls* - Two steel balls, 9.5 mm (3/3 in.) in diameter, each having a mass of 3.50 ± 0.05 g.
- *Ball-Centering Guides* - Two brass guides for centering the steel balls, on for each ring.
- *Bath* - A glass vessel, capable of being heated, not less than 85 mm in inside diameter and not less than 120 mm in depth from the bottom of the flare.
- *Ring Holder and Assembly* - A brass holder designed to support the two rings in a horizontal position.
- *Thermometers* - An ASTM Low Softening Point Thermometer, having a range from -2 to +80 °C.

Preparation of Test Specimen:

1. Do not start unless it is planned to complete preparation and testing of all asphalt specimens within 6 h.
2. Heat bitumen sample with care, stirring frequently to prevent local overheating, until it has become sufficiently fluid to pour.
3. Stir carefully to avoid incorporation of air bubbles in the sample.

4. Take no more than 2 h to heat an asphalt sample to its pouring temperature; in no case shall this be more than 110 °C above the expected softening point of the asphalt.
5. Heat the two brass rings (but not the pouring plate) to the approximate pouring temperature, and place them on the pouring plate treated with one of the release agents.
6. Pour a slight excess of the heated bitumen into each ring, and then allow the specimens to cool in ambient air for at least 30 min.
7. When the specimens have cooled, cut away the excess bitumen cleanly with a slightly heated knife or spatula, so that each disk is flush and level with the top of its ring.

Test Procedure:

1. Take freshly boiled distilled water. The starting bath temperature shall be 5 ± 1 °C.
2. Assemble the apparatus with the specimen rings, ball-centering guides, and thermometer in position, and fill the bath so that the liquid depth will be 105 ± 3 mm with the apparatus in place.
3. Place the bath in the ice water, and using forceps, place the two steel balls in the bottom of the bath and maintain the starting temperature of 5 ± 1 °C for 15 min.
4. Again using forceps, place a ball from the bottom of the bath in each ball-centering guide.
5. Heat the bath from below so that the temperature indicated by the thermometer rises at a uniform rate of 5 °C . The maximum permissible variation for any 1-min. period after the first 3 min shall be ± 0.5 °C.
6. Record for each ring and ball the temperature indicated by the thermometer at the instant the bitumen surrounding the ball touches the bottom plate.

Experimental Data and Results: Tabulate the data in the given *Test Data Sheet.* When using ASTM Thermometer, report to the nearest 0.2 °C.

The single-operator standard deviation has been found to be 0.41 °C. Therefore, results of two properly conducted tests by the same operator on the same sample of bitumen should not differ by more than 1.2 °C.

Discussion: Discuss about the results in the given *Test Data Sheet.*
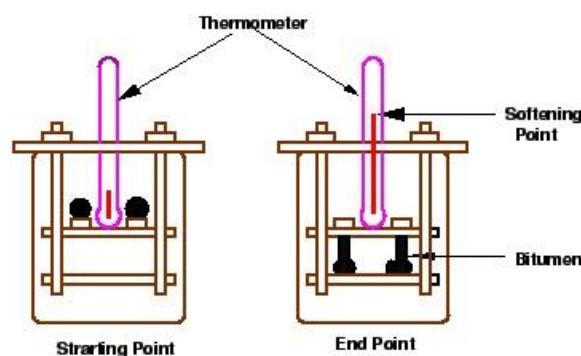
Experiment No. 2
Test Data Sheet
SOFTENING POINT OF BITUMEN

Sample No. _____ Description of Material: _____
Tested by: _____ Date of Testing: _____
Test Temperature: _____ °C Rate of Heat: _____ °C

Ring No.	Instant Temperature, °C	Softening Point, Average °C
1		
2		

Ring No.	Instant Temperature, °C	Softening Point, Average °C
1		
2		

Ring No.	Instant Temperature, °C	Softening Point, Average °C
1		
2		

Discussion of Test Results:

_____

_____

_____

_____

_____

_____

_____

_____

Standard Test Method For
FLASH AND FIRE POINTS BY CLEVELAND OPEN CUP

Objective: This test method covers determination of the flash and fire point of all petroleum products except fuel oils and those having an open flash cup below 79 °C.

Significance and Use:

- The flash point is one measure of the tendency of the test specimen to form a flammable mixture with air under controlled laboratory condition.
- Flash point is used in shipping and safety regulations to define flammable and combustible materials.
- The fire point is one measure of the tendency of the test specimen to support combustion.

Relevant Theory: The flash point of an asphalt material is the temperature at which its vapors will ignite instantaneously in the presence of an open flame. Note that the flash point normally is lower than the temperature at which the material will burn.

Standard Reference: ASTM D 92 – 90 and AASHTO T48 – 06

Apparatus Required:

- *Cleveland Open Cup Apparatus* - This apparatus consists of the test cup, heating plate, test flame applicator, heater, and supports.
- *Shield* - A shield 18 in. square and 24 in. high and having an open front is recommended.
- *Thermometer* - A thermometer having a range of -6 to 400 °C.

Sampling:

- Do not store samples in plastic containers
- Do not use samples from leaky containers.

Test Procedure:

1. Fill the cup at any convenient temperature so that the top of meniscus is exactly at the filling line. Remove any air bubbles, light the test flame and adjust it to the size of the comparison head on the apparatus.
2. Apply heat initially so that the rate of temperature rise of sample is 14 - 17 °C / min. When the sample temperature is approximately 56 °C below the anticipated flash point decrease the heat so that the rate of temperature rise is 5 - 6 °C / min.
3. Starting at least 28 °C below flash point apply test flame across the center of cup with a smooth continuous motion. The time consumed in passing the test flame across the cup in each case is 1 second.

4. Record as the observed flash point the temperature read on the thermometer when a flash appears at any point on the surface of the asphalt, but does not confuse the true flash with the bluish halo that sometimes surrounds the test flame.
5. Continue heating to determine the fire point so that the sample temperature increases at a rate of 5 - 6 °C / min. Continue the application of the test flame at 2 °C interval until the asphalt ignites and continues to burn at least 5 seconds.
6. Record the temperature at this point as the observed fire point of the asphalt.

Experimental Data and Results: Tabulate the data in the given *Test Data Sheet*. Values should be rounded off to the nearest 2 °C when reported.

Discussion: Discuss about the test and results in the given *Test Data Sheet.*

Apparatus for Cleveland Open-Cup Test

Experiment No. 3
Test Data Sheet
FLASH AND FIRE POINTS BY CLEVELAND OPEN CUP

Sample No. _____   Description of Material: _____

Tested by: _____   Date of Testing: _____

Test No.	Flash Point, °C	Fire Point, °C
1		
2		
3		
Average		

Discussion of Test Results:

_____

_____

_____

_____

_____

_____

_____

_____

Standard Method of Test for
DUCTILITY OF BITUMINOUS MATERIALS

Objective: The ductility of bituminous material is measured by the distance to which it will elongate before breaking when two ends of a briquette specimen of the material; are pulled apart at a specified speed and at a specified temperature. Unless otherwise specified, the test shall be made at a temperature of 25 ± 0.5 °C (77 ± 0.9 °F) and with a speed of 5 cm per minute plus or minus 5.0 %.

Significance and Use: This test method provides one measure of tensile properties of bituminous materials and may be used to measure ductility for specification requirements.

Relevant Theory: Ductility is the distance in centimeters a standard sample of asphalt material will stretch before breaking when tested on standard ductility test equipment. The result of this test indicates the extent to which the material can be deformed without breaking. This is an important characteristic for asphalt materials, although the exact value of ductility is not as important as the existence or nonexistence of the property in the material.

Standard Reference: ASTM D 113 and AASHTO T51

Apparatus Required:

- *Mold* - The mold shall be similar in design to that shown in ASTM manual.
- *Water Bath* - The water bath shall be maintained at the specified test temperature.
- *Testing Machine* - For pulling the briquette of bituminous material apart, any apparatus may be used which is so constructed that the specimen will be continuously immersed in water.
- *Thermometer* - A thermometer having a range of -8 to 32 °C.

Procedure Molding Test Specimen: The bituminous material to be tested shall be completely melted until thoroughly fluid by heating it in an oil bath maintained at the minimum temperature needed to properly liquefy the sample. It shall then be thoroughly stirred. The mold shall be assembled on a brass plate, to prevent the material under test from sticking the surface of the plate and interior surfaces of the sides of the mold shall be thoroughly amalgamated. In filling the mold, care should be taken not to damage the parts and thus distort the briquette. In filling, the material shall be poured in another stream back and forth from end to end of the mold to make sure it is more than level full. It shall be left to cool to room temperature for a period of 30 - 40 minutes. Then place it in the water bath maintained at the specified temperature of test for 30 minutes. Then the excess bitumen shall be cut off by means of a hot straight-edged putty knife or spatula so that the mold shall be just level full.

The brass place and mold, with briquette specimen, shall then be placed in the water bath and kept at the specified temperature for a period of from 85 - 95 minutes. When the briquette shall be removed from the plate, the side pieces detached, and the briquette immediately tested.

Testing: The rings at each end of the clips shall be attached at a pine or hooks in the testing machine. Then the two molds shall be pulled apart at a uniform speed and specified until the briquette ruptures. A variation of ± 5% from the speed specified will be permissible. The

distance through which the clips have been pulled to produce rupture shall then be measured in centimeters. While the test is being made, the water in the tank of the testing machine shall cover the specimen both above and below it by at least 2.5 cm. It shall be kept continuously covered at the temperature specified within ± 0.5 °C.

Experimental Data and Results: Tabulate the data in the given *Test Data Sheet.* A normal test is one in which the material between the two clips pull out to a point or thread until rupture occurs at the point where the thread has practically no cross-sectional area. The average of three normal tests shall be taken and reported as the ductility of the sample.
Adjust the specific gravity of the water by addition of either methyl alcohol or sodium chloride so that the bituminous material comes to the surface of neither water nor touches the bottom of the bath at any time during test. If the bituminous material comes in contact with the surface of water or the bottom of the bath, the test shall not be considered normal.

Discussion: Discuss about the test and results in the given *Test Data Sheet.*

Apparatus for Ductility Test

Experiment No.4
Test Data Sheet
DUCTILITY OF BITUMINIOUS MATERIALS

Sample No. _____   Description of Material: _____

Tested by: _____   Date of Testing: _____

Test Temperature: _____°C   Speed of pull: _____

Mold No.	Ductility reading, cms	Ductility average, cms
1		
2		
3		

Discussion of Test Results:

_____

_____

_____

_____

_____

_____

_____

_____

Experiment No. 5
Standard Method of Test for
VISCOSITY DETERMINATIONS OF UNFILLED ASPHALTS USING THE
BROOKFIELD THERMOSEL APPARATUS

Objective: This test method outlines a procedure for measuring the apparent viscosity of asphalt from 38 to 260°C (100 to 500°F) using the Brookfield Thermosel apparatus.

Significance and Use: This test method can be used to measure the apparent viscosity of asphalts at application temperatures. Some asphalts may exhibit non-Newtonian behavior under the conditions of this test, or at temperatures within the range of this method. Since non-Newtonian viscosity values are not unique material properties but reflect the behavior of the fluid and the measurement system, it should be recognized that measurements made by this method may not always predict performance under the conditions of use.

Relevant Theory: The Brookfield Thermosel Viscometer described in this procedure can be used to measure the viscosity of asphalt at elevated temperatures. The torque on a spindle rotating in a special thermostatically controlled sample holder containing a small sample of asphalt is used to measure the relative resistance to rotation. A factor is applied to the torque dial reading to yield the viscosity of the asphalt in millipascal seconds.

Standard Reference: ASTM D 4402 – 87

Apparatus Required:

- *Brookfield Thermosel High Temperature Viscosity Measurement System* - Using a Standard Brookfield Synchro-Lectric. Viscometer—Depending on viscosity range Model LV, RV, HA, or HB series may be used.
- *Spindles* - for Brookfield Thermosel Viscometer.
- *Thermosel System:*
  - Thermo Container and Sample Chamber.
  - SCR Controller and Probe.
  - Graph Plotting Equipment.

Test Procedure:

1. Read and understand the information in the instrument manufacturer's operating instructions before proceeding.
2. Turn on Thermosel power.
3. Set the proportional temperature controller to desired test temperature.
4. Refer to the operating instructions for calibration of the controller.
5. Wait 1.5 h (or until equilibrium temperature is obtained) with the selected spindle in the chamber (check control lamp).
6. Remove sample holder and add the volume of sample specified for the spindle to be used. Exercise caution to avoid sample overheating and to avoid ignition of sample with low flash point. Calculate the mass required from specific gravity or density data for the sample. Approximately 8 to 10 mL will be required.

7. Do not overfill the sample container. The sample volume is critical to meet the system calibration standard. Thoroughly stir filled asphalt coatings to obtain a representative sample.
8. The liquid level should intersect the spindle shaft at a point approximately 3.2 mm (1⁄8in.) above the upper "conical body"—"spindle shaft" interface.
9. Using the extracting tool, put the loaded chamber back into the thermo container.
10. Lower the viscometer and align the thermo-container.
11. Insert the selected spindle into the liquid in the chamber, and couple it to the viscometer. Proper spindle selection may require testing with more than one spindle.
12. Allow the asphalt to come to the equilibrium temperature (about 15 min)
13. Start Brookfield models RV, HA, HB viscometer at 20 rpm, LV model at 12 rpm. Observe the meter reading. If it is between 2 and 98 units, proceed with the test.
14. Record three readings 60 s apart at each test temperature.
15. Follow the procedure for each test temperature required.
16. If readings are above 98 units at the lowest test temperature, decrease the spindle rpm setting and continue with the test.
17. If the reading is above 98, use the next smaller spindle and repeat the procedure using the sample volume specified.
18. Multiply the viscosity factor by the Brookfield reading to obtain viscosity in centipoise.
19. Do not change the speed (rpm setting) during a viscosity measurement, as this will change the shear rate.

Experimental Data and Results: Tabulate the data in the given *Test Data Sheet.* Report test temperature, spindle number, and speed with results. For example, viscosity at 60°C = 105 mPa with spindle number. Plot viscosity value versus actual test temperature for each of the three or more test temperatures and draw a curve.

Discussion: Discuss about the test and results in the given *Test Data Sheet.*

Experiment No.5
Test Data Sheet
VISCOSITY DETERMINATIONS OF UNFILLED ASPHALTS USING THE BROOKFIELD THERMOSEL APPARATUS

Sample No. _____   Description of Material: _____

Tested by: _____   Date of Testing: _____

Test Temperature: _____°C

Mold No.	Viscosity reading, millipascal seconds (mPa·s)	Viscosity average, millipascal seconds (mPa·s)
1		
2		
3		

Discussion of Test Results:

_____
_____
_____
_____
_____
_____
_____
_____

Experiment No. 6
Standard Test Methods and Practices for
EMULSIFIED ASPHALTS: WATER CONTENT

<u>Objective</u>: This test method covers the procedure for determining the water content of an emulsified asphalt by reflux distillation using a water trap.

<u>Significance and Use</u>: This test method measures the amount of water present in the emulsified asphalt, as distinguished from either bitumen or petroleum solvent.

<u>Relevant Theory</u>:

<u>Standard Reference</u>: ASTM D244 -04

<u>Apparatus Required</u>:

- *Metal Still* - The metal still (Fig. 1(a)) shall be a vertical cylindrical vessel, preferably of copper, having a faced flange at the top to which the head is tightly attached by means of a clamp. The head shall be made of metal, preferably brass or copper, and shall be provided with a tubulation 25.4 mm (1 in.) in inside diameter.
- *Glass Still* - The glass still (Fig. 1(b)) shall be a shortneck, round-bottom flask, made of well-annealed glass, and having an approximate capacity of 500 mL.
- *Heat Source* - The heat source used with the metal still shall be a ring gas burner of 100-mm (4-in.) inside diameter or an electric mantle heater. The heat source for the glass still shall be either an ordinary gas burner or an electric heater.
- *Condenser* - The condenser shall be a water-cooled reflux glass-tube type, having a jacket not less than 400 mm in length, with an inner tube 9.5 to 12.7 mm in outside diameter. The end of the condenser shall be ground to an angle of $30 \pm 5°$ from the vertical axis of the condenser.
- *Trap* - The trap shall be made of annealed glass constructed in accordance with Fig. 1(c) and shall be graduated in
- 0.10-mL divisions from 0 to 2 mL, and in 0.20-mL divisions from 2 to 25 mL.
- *Solvent* - Xylol or other petroleum distillate conforming to the following distillation requirements: 98 % distills between 120 and 250°C. This distillation shall be conducted in accordance with Test Method D 86.
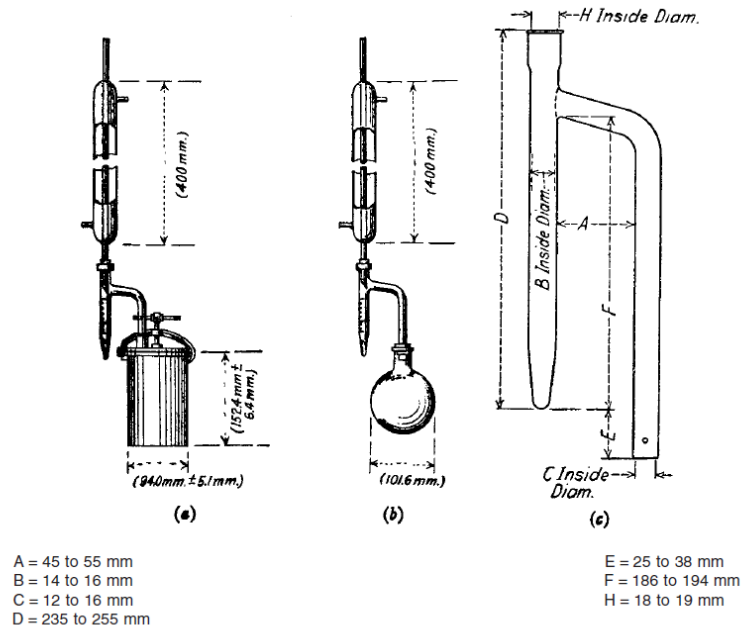
A = 45 to 55 mm   E = 25 to 38 mm
B = 14 to 16 mm   F = 186 to 194 mm
C = 12 to 16 mm   H = 18 to 19 mm
D = 235 to 255 mm

FIG. 1 Apparatus for Determining Water Content

Test Procedure:

1. When the material to be tested contains less than 25 % water, place 100 ± 0.1 g of sample in the still. When the material contains more than 25 % water, use a 50 ± 0.1-g sample. Thoroughly mix the sample to be tested with 200 mL of solvent by swirling, taking proper care to avoid any loss of material.
2. Connect the still, trap, and condenser by means of tight-fitting corks as shown in Fig. 1(a) or (b). Adjust the end of the condenser in the trap to a position which will allow the end to be submerged to a depth of not more than 1 mm below the surface of the liquid in the trap after distillation conditions have been established. When using the metal still, insert a heavy paper gasket, moistened with the solvent, between the lid and flange before attaching the clamp.
3. When the ring burner is used with the metal still, place it about 76.2 mm above the bottom of the still at the beginning of the distillation, and gradually lower it as the distillation proceeds. Regulate the heat so that the condensate falls from the end of the condenser at a rate of from 2 to 5 drops per second. Continue the distillation at the specified rate until no water is visible on any part of the apparatus and a constant volume of water is obtained in the trap. Remove any persistent ring of condensed water in the condenser tube by increasing the rate of distillation for a few minutes.

Experimental Data and Results: Calculate the water content as follows:

Water content, % = (A/B) × 100
Where:
A = volume of water in trap, mL, and
B = original weight of sample, g.

Discussion: Report the result as "…... water weight percent, ASTM D 244."

Experiment No. 7
Marshall Mix Design
Part I
ASPHALT PAVING MIX DESIGN

<u>Objective:</u> The design of asphalt paving mixes is largely a matter of selecting and proportioning materials to obtain the desired properties in the finished construction. The overall objective of the design of asphalt paving mixes is to determine an economical blend and gradation of aggregates and asphalt that yields a mix having:

- Sufficient asphalt to ensure a durable pavement
- Sufficient mix stability to satisfy the demands of traffic without distortion or displacement
- Sufficient voids in the total compacted mix to allow for a slight amount of additional compaction under traffic loading without flushing, bleeding, and loss of stability
- Sufficient workability to permit efficient placement of the mix without segregation.

<u>Relevant Theory of Marshall Method of Mix Design:</u>

Outline of Method:

1. The materials should meet the project specifications.
2. The aggregate gradation should not meet the project specifications.
3. The bulk specific gravity of aggregates and the specific gravity of the asphalt cement are determined.

General details:

- Only applicable for hot-mix paving mixture
- Asphalt cement have to be used
- Maximum size of aggregate = 1" (25 mm) or less
- Standard test specimens are 2.5" high by 4" diameter
- Two principles features of the method:
    - density - voids analysis
    - stability - flow test of the compacted test specimens
- The stability of the specimen is the maximum load resistance in pounds which the specimen can carry at 140 °F (60 °C)
- The flow value is the total movement or strain in units of 1/100 in. (0.25 mm) occurring in the specimen between no load and maximum load during the stability test
- For preparation of specimens:
    - Aggregate weight used is 1200 g
    - Aggregate should be heated to 160 °C for 3 to 4 h
    - Asphalt cement should also be heated to 155 °C for not more than one hour to prevent over heating
- Mixing temperature is (155 °C) which produce kinematic viscosity of 170 ± 20 cst
- Compaction temperature is (145 °C) which produce kinematic viscosity of 280 ± 30 cst
- The molds, hammer should be heated between 93 °C (200 °F) and 149 °C (300 °F)
- Use 75 blows on each side of the specimen to compact the specimens for heaving traffic (hammer weight = 10 pound and drop = 18 in)

- Specimens are allowed to cool overnight
- Trial specimen is prepared:

  Adjusted Wt. of Agg. = 2.5 (Wt. of Agg. used) ÷ Spec. Ht. (in) obtained
  (U.S. Units)
  AdjustedWt.of Agg .= 63.5 (Wt. of Agg. used) ÷ Spec. Ht. (mm) obtained
  (SI Units)

In the Marshall method, each compacted test specimen is subjected to the following tests and analysis in the order listed:

  - Bulk Specific Gravity Determination
  - Stability - Flow Test
  - Density & Voids Analysis

TABLE 7.1 - Marshall Design Criteria

Marshall Method Mix Criteria	Light Traffic Surface & Base		Medium Traffic Surface & Base		Heavy Traffic Surface & Base	
	Min	Max	Min	Max	Min	Max
Compaction, number of blows each end of specimen	35		50		75	
Stability, N (lb)	3336 (750)	-	5338 (1200)	-	8006 (1800)	-
Flow, 0.25 mm	8	18	8	16	8	14
Percent Air Voids	3	5	3	5	3	5
Percent Voids in Mineral Aggregate (VMA)			See Figure 16-1			
Percent Voids Filled With Asphalt (VFA)	70	80	65	78	65	75

Experiment No. 7-A
Part II
Standard Test Method For
RESISTANCE TO PLASTIC FLOW OF BITUMINOUS MIXTURES USING
MARSHALL APPARATUS

Objective: This test method covers the measurement of the resistance to plastic flow of cylindrical specimens of bituminous paving mixture loaded on the lateral surface by means of the Marshall apparatus. This test method is for use with mixtures containing asphalt cement, asphalt cut-back or tar, and aggregate up to 1-in. (25.4-mm) maximum size.

Standard Reference: ASTM D 1559 – 89

Apparatus Required:

- *Specimen Mold Assembly -* Mold cylinders 4 in. in diameter by 3 in. in height, base plates, and extension collars
- *Specimen Extractor,* steel, in the form of a disk with a diameter not less than 3.95 in. and ½ in. thick for extracting the compacted specimen from the specimen mold with the use of the mold collar.
- *Compaction Hammer -* The compaction hammer shall have a flat, circular tamping face and a 10-lb sliding weight with a free fall of 18 in.
- *Compaction Pedestal -* The compaction pedestal shall consist of an 8 by 8 by 18-in. wooden post capped with a 12 by 12 by 1-in. steel plate.
- *Specimen Mold Holder,* mounted on the compaction pedestal to center the compaction mold over the center of the post. It shall hold the compaction mold, collar, and base place securely in position during compaction of the specimen.
- *Breaking Head -* The breaking head shall consist of upper and lower cylindrical segments or test heads having an inside radius of curvature of 2 in.
- *Loading Jack -* The loading jack shall consist of a screw jack mounted in a testing frame and shall produce a uniform vertical movement of 2 in. / min.
- *Ovens or Hot Plates*
- *Mixing Apparatus -* Mechanical mixing is recommended.
- *Water Bath -* The water bath shall be at least 6 in. deep and shall be thermostatically controlled so as to maintain the bath at 140 ± 1.8 °F (60 ± 1.0 °C). The tank shall have a perforated false bottom or be equipped with a shelf for supporting specimens 2 in. above the bottom of the bath.
- *Miscellaneous Equipment:*
- *Containers*
- *Mixing Tool*
- *Thermometers*
- *Balance*
- *Gloves*
- *Rubber Gloves*
- *Marking Crayons*
- *Scoop*
- *Spoon*

Preparation of Test Specimens:

- *Number of Specimens* - Prepare at least three specimens for each combination of aggregates and bitumen content
- *Preparation of Aggregates* - Dry aggregates to constant weight at 221 to 230 °F (105 to 110 °C) and separate the aggregates to dry sieving into the desired size fractions.
- *Determination of Mixing and Compacting Temperatures:*
- The temperatures to which the asphalt cement must be heated to produce a viscosity of 170 ±20 cSt shall be the mixing temperature.
- The temperature to which asphalt cement must be heated to produce a viscosity of 280 ± 30 cSt shall be the compacting temperature.
- *Preparation of Mixtures:* Weigh into separate pans for each test specimen the amount of each size fraction required to produce a batch that will result in a compacted specimen 2.5 ± 0.05 in. in height (about 1200 g). Place the pans on the hot plate or in the oven and heat to a temperature not exceeding the mixing temperature by more than approximately 28 °C . Charge the mixing bowl with the heated aggregate and dry mix thoroughly. Form a crater in the dry blended aggregate and weigh the preheated required amount of bituminous material into the mixture. Mix the aggregate and bituminous material rapidly until thoroughly coated.
- *Compaction of Specimens:*
- Thoroughly clean the specimen mold assembly and the face of the compaction hammer and heat them either in boiling water or on the hot plate to a temperature between 200 and 300 °F (93.3 and 148.9 °C). Place a piece of filter paper or paper toweling cut to size in the bottom of the mold before the mixture is introduced. Place the entire batch in the mold, spade the mixture vigorously with a heated spatula or trowel 15 times around the perimeter and 10 times over the interior. Remove the collar and smooth the surface of the mix with a trowel to a slightly rounded shape.

Replace the collar, place the mold assembly on the compaction pedestal in the mold holder, and apply 75 blows with the compaction hammer with a free fall in 18 in. Remove the base plate and collar, and reverse and reassemble the mold. Apply the same number of compaction blows to the face of the reversed specimen. After compaction, remove the base plate and place the sample extractor on that end of the specimen. Place the assembly with the extension collar up in the testing machine, apply pressure to the collar by means by means of the load transfer bar, and force the specimen into the extension collar. Lift the collar from the specimen. Carefully transfer the specimen to a smooth, flat surface and allow it to stand overnight at room temperature. Weigh, measure, and test the specimen.

Testing Procedure:

1. Bring the specimens prepared with asphalt cement to the specified temperature by immersing in the water bath 30 to 40 min or placing in the oven for 2 h. Maintain the bath or oven temperature at 140 ± 1.8 °F (60 ± 1.0 °C) for the asphalt cement specimens. Thoroughly clean the guide rods and the inside surfaces of the test heads prior to making the test, and lubricate the guide rods so that the upper test head slides freely over them. The testing head temperature shall be maintained between 70 to 100 °F (21.1 to 37.8 °C) using a water bath. Remove the specimen from the water bath, oven, or air bath, and place in the lower segment of the breaking head. Place the upper

segment of the breaking head on the specimen, and place the complete assembly in position on the testing machine.

2. Apply the load to the specimen by means of the constant rate of movement of the load jack or testing machine head of 2 in./min until the maximum load is reached and the load decreases as indicated by the dial. Record the maximum load and the indicated flow. The elapsed time for the test from removal of the test specimen from the water bath to the maximum load determination shall not exceed 30 s.

TABLE 7.2 Stability Correlation Ratios[A]

Volume of Specimen, cm^3	Approximate Thickness of Specimen, in.[B]	mm	Correlation Ratio
406 to 420	2	50.8	1.47
421 to 431	2 1/16	52.4	1.39
432 to 443	2 1/8	54.0	1.32
444 to 456	2 3/16	55.6	1.25
457 to 470	2 1/4	57.2	1.19
471 to 482	2 5/16	58.7	1.14
483 to 495	2 3/8	60.3	1.09
496 to 508	2 7/16	61.9	1.04
509 to 522	2 ½	63.5	1
523 to 535	2 9/16	65.1	0.96
536 to 546	2 5/8	66.7	0.93
547 to 559	2 11/16	68.3	0.89
560 to 573	2 3/4	69.8	0.86
574 to 585	2 13/16	71.4	0.83
586 to 598	2 7/8	73.0	0.81
599 to 610	2 15/16	74.6	0.78
611 to 625	3	76.2	0.76

[A]The measured stability of a specimen multiplied by the ratio for the thickness of the specimen equals the corrected stability for a 2 1/2 in. (63.5 mm) specimen.

[B]Volume-thickness relationship is based on a specimen diameter of 4 in. (101.6 mm).

Experimental Data and Results:

- Tabulate the test data in the given *Test Data Sheet*. Report the type of sample tested (laboratory sample or pavement core specimen)
- Average maximum load in pounds-force of at least three specimens, corrected when required.
- Average flow value, in hundredths of an inch, twenty-five hundredths of a millimeter, of three specimens,
- Test temperature.

TABLE 7.3: BITUMINOUS WEARING COURSE
Optimum Blending for Marshall Mix Design
(M.O.C. Class "A" Aggregate Blending)

Sieve Size	% Individual	Individual Weights g	Cumulative Weights g	Specification Limits % Passing	% Mean	Tolerance
3/4"	0.0	0	0	100	100	± 6
1/2"	16.0	192	192	76 - 92	84	± 6
3/8"	12.5	150	342	64 - 79	71.5	± 5
# 4	23.0	276	618	41 - 56	48.5	± 5
#10	10.5	222	840	23 - 37	30	± 4
#40	16.5	198	1038	7 - 20	13.5	± 4
#80	4.5	54	1092	5 - 13	9	± 3
#200	3.5	42	1134	3 - 8	5.5	± 1.5
Filler	5.5	66	1200	-	-	

Coarse Aggregate,% = Passing 3/4" to Retained # 4
 = 51.5 %

Fine Aggregate, % = Passing # 4 to Retained # 200
 = 43 %

Filler, % = Passing # 200
 = 5.5 %

Total Aggregate % 100.0

Experiment 7-B
Part III
Standard Method of Test For
THEORETICAL MAXIMUM SPECIFIC GRAVITY AND DENSITY OF BITUMINOUS PAVING MIXTURES

Objective: This test method covers the determination of the theoretical maximum specific gravity and density of uncompacted bituminous paving mixtures at 25 °C

Summary of Test Method: A weighed sample of oven-dry paving mixture in the loose condition is placed in a tared vacuum vessel. Sufficient water at a temperature of 25 ± 4 °C is added to completely submerge the sample. Vacuum is applied for from 5 to 15 min to gradually reduce the residual pressure in the vacuum vessel to 30 mm Hg or less. At the end of the vacuum period, the vacuum is gradually released. The volume of the sample of paving mixture is obtained by filling the vacuum container level full of water and weighing in air. At the time of weighing, the temperature is measured as well as the mass. From the mass and volume measurements, the specific gravity or density at 25 °C is calculated. If the temperature employed is different from 25 °C, an appropriate correction is applied.

Standard Reference: ASTM D 2041-91

Apparatus Required:

- *Vacuum Container* - A small volumetric flask with a capacity of approximately 2000 mL.
- *Balance* - with ample capacity, and with sufficient sensitivity to enable the specific gravity of samples of uncompacted paving mixtures to be calculated to at least four significant figures.
- *Vacuum pump or water aspirator,* capable of evacuating air from the vacuum container to a residual pressure of 30 mm Hg.
- *Residual pressure manometer* - To be connected directly to the vacuum vessel and to be capable of measuring residual pressure down to 30 mm of Hg.
- *Manometer or Vacuum Gauge* - Suitable for measuring the vacuum being applied at the source of the vacuum.
- *Thermometers* - Calibrated liquid-in-glass thermometers of suitable range.

Sampling: Obtain the sample according to the following size requirements:

Size of Largest Particle of Aggregate:

Mixture, in.	Minimum Sample Size, g
1 ½	4000
1	2500
¾	2000
½	1500
¼	1000
No. 4	500

Calibration of Flask:

- Calibrate the flask by determining the mass of the flask when filled with water over the range of water temperatures likely to be encountered in service. Designate this mass as *D*. Plot a graph of mass of flask filled with water versus water temperatures.

Procedure:

1. Separate the particles of the sample of paving mixture by hand, taking care to avoid fracturing the aggregate, so that the particles of the fine aggregate portion are not larger than 1/4 in. If a sample of paving mixture is not sufficiently soft to be separated manually, place it is a flat pan, and warm it in an oven until it can be separated.
2. Unless the paving mixture has been prepared in a laboratory using oven-dry aggregates, oven-dry the sample to constant mass at a temperature of 105 ± 5 °C.
3. Cool the sample to room temperature, place it in a tare container and weigh. Designate the net mass of the sample as *A*. Add sufficient water at a temperature of approximately 25 °C to cover the sample completely.
4. Remove air trapped in the sample by applying gradually increased vacuum until the residual pressure manometer reads 30 mm Hg or less. Maintain this residual pressure for 5 to 15 min. Agitate the container and contents during the vacuum period either continuously by a mechanism device, or manually by vigorous shaking at intervals of about 2 min.
5. At the end of the vacuum period, gently release the vacuum and fill the flask with water and adjust the contents to a temperature of 25 ± 1 °C. Determine the mass of the container (and contents), completely filled. Designate this mass as *E*.

Experimental Data, Results and Calculations:

- Tabulate the data in the given *Test Data Sheet*. Report the theoretical maximum specific gravity to the nearest third decimal.
- Calculate the theoretical maximum specific gravity of the sample at 25 °C as follows:

$$\text{Theoretical Maximum Specific Gravity} = A / (A + D - E)$$
Where:

A = mass of oven dry sample in air, g
D = mass of container filled with water at 25 °C
E = mass of container filled with sample and water at 25 °C

Discussion: Discuss about the test and results in the given *Test Data Sheet*.

Experiment No. 7-B
Test Data Sheet
THEORETICAL MAXIMUM SPECIFIC GRAVITY OF
LOOSE PAVING MIXTURE

Sample No. _____ Description of Material: _____

Tested by: _____ Date of Testing: _____

Test Temperature: _____ ° C;   Sample Type: _____

Test No.	Mass of Oven Dry Sample in Air, g A	Mass of Container filled with water at 25 °C D	Mass of Container filled with sample and water at 25 °C E	Theoretical Maximum Specific Gravity $A / (A+D-E)$
1				
2				
3				
Average Theoretical Maximum Specific Gravity				

Discussion of Test Results: _____

_____

_____

_____

_____

Experiment No. 7-C
Part IV
ANALYSIS OF COMPACTED PAVING MIXTURES

General: The analytical procedures described herein apply either to paving mixtures that have been compacted in the laboratory, or to undisturbed samples that have been cut from a pavement in the field. When a paving mixture is compacted in the laboratory, the compactive effort should provide a density equal to the density the mixture will ultimately attain under traffic following compaction by rolling during construction.

By analyzing a compacted paving mixture (V_a), voids in the mineral aggregate (VMA), and effective asphalt content (P_{be}), some indication of the probable service performance of the pavement is provided. The efficacy of compaction, either during construction or after years of service can be determined by comparing the specific gravity of an undisturbed sample cut from a pavement with the laboratory compacted specific gravity of the paving mixture.

Definition:

- **Bulk Specific Gravity (G_{sb}):** The ratio of the weight in air of a unit volume of a permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of equal volume of gas free distilled water at a stated temperature. Refer to Figure 18-3.
- **Apparent Specific Gravity (G_{sa}):** The ratio of the weight in air of a ;unit volume of an impermeable material at a stated temperature to the weight in air of equal density of an equal volume of gas free distilled water at a stated temperature. Refer to Figure 16-3.
- **Effective Specific Gravity (G_{se}):** The ratio of the weight in air of a unit volume of a permeable material (excluding voids permeable to asphalt) at a stated temperature to the weight in air of equal density of an equal volume of gas free distilled water at a stated temperature. Refer to Figure 18-3.
- **Voids in the Mineral Aggregate (VMA):** The volume of intergranular void space between the aggregate particles of a compacted paving mixture that includes the voids and the effective asphalt content, expressed as a percent of the total volume of the sample. Refer to Figure 18-3.
- **Effective Asphalt Content (P_{be}):** The total asphalt content of a paving mixture minus the portion of asphalt that is lost by absorption into the aggregate particles. Refer to Figure 18-3.
- **Air Voids (V_a):** The total volume of the small pockets of air between the coated aggregate particles throughout a compacted paving mixture, expressed as percent of the bulk volume of the compacted paving mixture. Refer to Figure 18-3.
- **Volume Relationships in a Compacted Mix:** Figure 18-4 depicts the volume relationships between aggregate, air voids in mineral aggregate, total asphalt content, asphalt lost by absorption into the aggregate particles, and effective asphalt content.

OUTLINING PROCEDURE FOR ANALYZING A COMPACTED PAVING MIXTURE:

1. Measure the bulk specific gravities of the coarse aggregate and of the fine aggregate.
2. Measure the specific gravity of the asphalt cement and the mineral filler.
3. Calculate the bulk specific gravities of the aggregate combination in the paving mixture.
4. Measure the maximum specific gravity of the loose paving mixture. (ASTM D 2041)
5. Measure the bulk specific gravity of the compacted paving mixture.
6. Calculate the effective specific gravity of aggregate.
7. Calculate asphalt absorptions of aggregate.
8. Calculate the effective asphalt content of the paving mixture.
9. Calculate the percent voids in the mineral aggregate in the compacted paving mixture.
10. Calculate the percent air voids in the compacted paving mixture.

Paving Mixture Data for Sample Calculations

Material	Specific Gravity Standards		ASTM Method	Mix Composition
	Apparent	Bulk		Percent by Wt. of Total Mix
Asphalt Cement		-	D 70	P_b
Coarse Aggregate		G_1	C 127	P_1
Fine Aggregate		G_2	C 128	P_2
Mineral Filler	G_3	-	D 854	P_3

EQUATIONS FOR SAMPLE CALCULATIONS:

- **Bulk Specific Gravities of Aggregate:** When the total aggregate consists of separate fractions of coarse aggregate, fine aggregate, and mineral filler, all having different specific gravities

$$G_{sb} = \frac{P_1 + P_2 + ... + P_n}{\dfrac{P_1}{G_1} + \dfrac{P_2}{G_2} + ... + \dfrac{P}{G_n}}$$

  where:

  G_{sb} = bulk specific gravity for the total aggregate
  P_1 , P_2 , P_n = percentages by weight of aggregates 1, 2, ... n; and,
  G_1 , G_2 , G_n = bulk specific gravities of aggregate 1, 2, ... n

- **Effective Specific Gravity of Aggregate:** When based on the maximum specific of a paving mixture, G_{mm} , the effective specific gravity of G_{se} , of the aggregate includes all void spaces in the aggregate particles except those that absorb asphalt. It is determined as follows:

$$G_{se} = \frac{P_{mm} - P_b}{\dfrac{P_{mm}}{G_{mm}} - \dfrac{P_b}{G_b}}$$

  where:

  G_{se} = effective specific gravity of aggregate;

P_{mm} = total loose mixture, percent by total weight of mixture = 100 percent,

P_b = asphalt, percent by total weight of mixture,

G_{mm} = maximum specific gravity of paving mixture (no air voids), ASTM D 2041,

and

G_b = specific gravity of asphalt.

- **Maximum Specific Gravities of Mixtures with Different Asphalt Contents:** In designing a paving mixture with a given aggregate, the maximum specific gravities, G_{mm}, at different asphalt contents are needed to calculate the percentage of air voids for each asphalt content. While the same maximum specific gravity can be determined for each asphalt content by ASTM test method (D 2041).

  After getting the results from these tests and calculating the effective specific gravity of the aggregate, the maximum specific gravity for any other asphalt can be obtained as shown below. For all practical purposes, the effective specific gravity of the aggregate is constant because asphalt absorption does not vary appreciably with variation in asphalt content.

$$G_{mm} = \frac{P_{mm}}{\dfrac{P_s}{G_{se}} + \dfrac{P_b}{G_b}}$$

  where:

  G_{mm} = maximum specific gravity of paving mixture (no air voids)
  P_{mm} = total loose mixture, percent by total weight of mixture = 100 percent
  P_s = aggregate, percent by total weight of mixture
  P_b = asphalt percent by total weight of mixture
  G_{se} = effective specific gravity of aggregate, and
  G_b = specific gravity of asphalt

- **Asphalt Absorption:** Absorption is expressed as a percentage by weight of aggregate rather than as a percentage by total weight of mixture. Asphalt, P_{ba} absorption is determined as follows:

$$P_{ba} = 100 * \frac{G_{se} - G_{sb}}{G_{se} * G_{sb}} * G_b$$

  where:

  P_{ba} = absorbed asphalt, percent by weight of aggregate,
  G_{se} = effective specific gravity of aggregate,
  G_{sb} = bulk specific gravity of aggregate, and
  G_b = specific gravity of asphalt

- **Effective Asphalt Content of a Mixture:** The effective asphalt content, P_{be} of a paving mixture is the total asphalt content minus the quantity of asphalt lost by absorption into the aggregate particles. It is the portion of the total asphalt content that remains as a coating on the outside of the aggregate particles, and is the asphalt content on which service performance of an asphalt paving mixture depends. The formula is:

$$P_{ba} = P_b - \frac{P_{ba}}{100} * P_s$$

where:

P_{be} = effective asphalt content, percent by total weight of mixture,
P_b = asphalt, percent by total weight of mixture,
P_{be} = absorbed asphalt, percent by weight of aggregate, and,
P_s = aggregate, percent by total weight of mixture.

- **Percent VMA in Compacted Paving Mixture:** The voids in the mineral aggregate, VMA, are defined as the intergranular void space between the aggregate particles in a compacted paving mixture that includes the air voids and the effective asphalt content, expressed as a percent of the total volume. The VMA is calculated on the basis of the bulk specific gravity of the aggregate and is expressed as a percentage of the bulk volume of the compacted paving mixture.

$$VMA = 100 - \left(\frac{G_{mb} * P_s}{G_{sb}} \right)$$

where:

VMA = voids in mineral aggregate (percent of bulk volume),
G_{sb} = bulk specific gravity of aggregate,
G_{mb} = bulk specific gravity of compacted mixture, and,
P_s = aggregate, percent by total weight of mixture.

- **Calculating Percent Air Voids in Compacted Mixture:** The air voids P_a , in a compacted paving mixture consist of the smaller air spaces between the coated aggregate particles. The percentage of air voids in a compacted mixtures can be determined by the following equation:

$$V_a = 100 * \left(\frac{G_{mm} - G_{mb}}{G_{mm}} \right)$$

where:

V_a = air voids in a compacted mixture, percent of total volume,
G_{mm} = maximum specific gravity of paving mixture (as determined above) or as determined directly for a paving mixture by ASTM D 2041,

G_{mb} = bulk specific gravity of compacted mixture.

- **Voids Filled with Asphalt:** The voids in the aggregate filled with asphalt, VFA, are defined as, the amount of voids in the aggregate of a compacted bituminous mixture is equal to the apparent volume of the mixture minus the true volume of the mineral aggregate. The percentage of the voids in the aggregate are calculated as follows:

$$VFA = \left(\frac{P_{be} * G_{mb}}{G_b * VMA} \right) * 100$$

where:

> VFA $=$ voids in the aggregate filled with asphalt,
> P_{be} $=$ effective asphalt content, percent by total weight of mixture,
> G_{mb} $=$ bulk specific gravity of compacted mixture,
> VMA $=$ voids in mineral aggregate (percent of bulk volume), and,
> G_b $=$ specific gravity of asphalt.

The voids in the aggregate filled with asphalt can also be calculated as:

$$VFA = \left(\frac{VMA - P_a}{VMA} \right) *100$$

where:

> P_a $=$ air voids in compacted mixture, percent of total volume,

- **Trends and Relations of Test Data:** The test property curves, plotted as described in Figure 18-5, should follow the pattern as described in the figure in a consistent form. Trends generally noted are outlined as follows:

1. The stability value increases with increasing asphalt content up to a maximum after which the stability decreases.
2. The follow value increases with increasing asphalt content.
3. The curve for unit weight of total mix is similar to the stability curve, except that the maximum unit weight normally (but not always) occurs at a slightly higher asphalt content than the maximum stability.
4. The percent of air voids decreases with increasing asphalt content, ultimately approaching a minimum void content.
5. The percent voids in the mineral aggregate generally decrease to a minimum value then increase with increasing asphalt contents.
6. The percent voids in the aggregate filled with asphalt rises rapidly at low binder contents and tends to level off at high binder contents.

- **Graphical Charts:**

   Prepare a graphical plot for the following values from the data obtained:
   1. Stability verses asphalt content
   2. Flow verses asphalt content
   3. Specific gravity of total mix verses
   4. Percent Air voids verses asphalt content
   5. Percent Voids in Mineral Aggregate (VMA) versus asphalt content
   6. Voids Filled with Asphalt (VFA) versus asphalt content

- **Specification Limits:**

Class A	Percent
Total Mineral Aggregate	96 - 93
Asphaltic Binder	4 - 7
Stability (kgs)	820 min.

Voids in total mix	3.0 - 5.0
Flow, 0.25 mm	8.0 - 14.0
Voids Filled with Asphalt (VFA), %	65 - 75
VMA, %	See Figure 16-4

Determination of Optimum Asphalt Content: The optimum asphalt content of the asphalt paving mix is determined from the test curves to be used for the medium traffic category. Compute asphalt content as follows:

Percent

1. Asphalt content at maximum specific gravity =
2. Asphalt content at maximum stability =
3. Asphalt content at 4 % Air Voids =
4. Asphalt content at 75 % V.F.A. =

_____

Optimum asphalt content, average _____

Experiment No. 8
Standard Method of Test for
QUANTITATIVE EXTRACTION OF BITUMEN FROM BITUMINOUS PAVING MIXTURES

Objective: These test methods cover the quantitative determination of bitumen in hot-mixed paving mixtures and pavement samples.

Significance and Use: All of these test methods can be used for quantitative determinations of bitumen in hot-mixed paving mixtures and pavement samples for specification acceptance, service evaluation, control, and research. Each method prescribes the solvent or solvents and any other reagents that can be used in the test method.

Relevant Theory: The paving mixture is extracted with trichloroethylene, normal Propyl Bromide, or methylene chloride using the extraction equipment applicable to the particular method. The bitumen content is calculated by difference from the mass of the extracted aggregate, moisture content, and mineral matter in the extract. The bitumen content is expressed as mass percent of moisture-free mixtures.

Standard Reference: ASTM D 2172 – 05

Apparatus Required:

- *Oven*, capable of maintaining the temperature at 230 ₆ 9°F (110 ₆ 5°C).
- *Pan*, flat, 12 in. (305 mm) long, 8 in. (203 mm) wide, and 1 in. (25 mm) deep.
- *Balance*, or balances having an accuracy of at least 0.01 % of the sample mass.
- *Hot Plate*, electric, 700-W continuous or low, medium, and high settings.
- *Small-Mouth Graduate*, 1000 or 2000-mL capacity. Optional small-mouth graduate, 100-mL capacity.
- *Ignition Dish*, 125-mL capacity.
- *Desiccator*.
- *Analytical Balance*.
- *Extraction Apparatus*, consisting of a bowl approximating and an apparatus in which the bowl may be revolved at controlled variable speeds up to 3600 r/min. The speed may be controlled manually or with a preset speed control. The apparatus should be provided with a container for catching the solvent thrown from the bowl and a drain for removing the solvent. The apparatus preferably shall be provided with explosion-proof features and installed in a hood or an effective surface exhaust system to provide ventilation.
- *Filter Rings*, felt or paper, to fit the rim of the bowl.
- Low-ash paper filter rings may be used in place of the felt filter ring. Such filter rings shall consist of low ash filter paper stock 0.05 ± 0.005 in. thick. The nominal base weight of the paper shall be 330 ± 30 lb for a ream (500 sheets—25 by 38 in.). The ash content of the paper should not exceed 0.2 % (approximately 0.034 g per ring).

Testing Condition: Where the conditions of test are not specifically mentioned, the temperature, load, and time are understood to be 25 °C, 100 g, and 5 s, respectively.

Test Procedure:

1. Determine the moisture content of the material.
2. Place a 650 to 2500-g test portion into a bowl.
3. Cover the test portion in the bowl with trichloroethylene, normal Propyl Bromide, or methylene chloride and allow sufficient time for the solvent to disintegrate the test portion (not over 1 h). Place the bowl containing the test portion and the solvent in the extraction apparatus. Dry and determine the mass of the filter ring and fit it around the edge of the bowl. Clamp the cover on the bowl tightly and place a beaker under the drain to collect the extract.
4. Start the centrifuge revolving slowly and gradually increase the speed to a maximum of 3600 r/min or until solvent ceases to flow from the drain. Allow the machine to stop, add 200 mL of trichloroethylene, normal Propyl Bromide, or methylene chloride and repeat the procedure. Use sufficient 200-mL solvent additions (not less than three) so that the extract is not darker than a light straw color. Collect the extract and the washings in a suitable graduate.
5. Remove the filter ring from the bowl and dry in air. If felt filter rings are used, brush off mineral matter adhering to the surface of the ring and add to the extracted aggregate. Dry the ring to constant mass in an oven at 230 6 9°F (110 6 5°C). Carefully remove all the contents of the bowl into a metal pan and dry on a steam bath and then, dry to constant mass in an oven or on a hot plate at 2306 9°F (110 6 5°C). If trichloroethylene or normal Propyl Bromide is used as the extraction solvent, the preliminary drying on a steam bath may be omitted. The mass of the extracted aggregate, W 3, is equal to the mass of the aggregate in the pan plus the increase in mass of the filter rings.
   - Use the following alternative procedure when low ash filter rings are used: Place the aggregate and filter rings in a clean metal pan. Dry as specified above. Carefully fold the dried filter ring and stand it on the aggregate. Burn the filter ring by igniting with a bunsen burner or match. Determine the mass of the extracted aggregate in the pan, W_3.
6. Determine the amount of mineral matter in the extract by any of the following test methods:

Ashing Method:

1. Record the volume of the total extract in the graduate (11.4). Determine the mass of an ignition dish. Agitate the extract thoroughly and immediately measure approximately 100 mL into the ignition dish. Evaporate to dryness on a steam bath or hot plate, except use a steam bath when the solvent is benzene. 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 h. Dry in an oven at 100°C to constant mass, cool in a desiccator, and determine the mass.
2. Calculate the mass of mineral matter in the total volume of extract, W_4, as follows:

$$W_4 = G[\frac{V_1}{V_1 - V_2}]$$

Where:
G = ash in aliquot, g,
V_1 = total volume, mL, and
V_2 = volume after removing aliquot, mL.

Centrifuge Method:

1. For this test method use any suitable high-speed (3000 g or higher) centrifuge of the continuous-flow type.
2. Determine the mass of a clean empty centrifuge cup (or bowl) to 0.01 6 0.005 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 (from Test Methods A, B, C, D, or E as appropriate) to an appropriate (feed) container suitably equipped with a feed control (valve or clamp, etc.). 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, 9000 r/min for the SMM type and 20 000 + r/min for the Sharples type). Open the feed line and feed the extract into the centrifuge at a rate of 100 to 150 mL/min. After all the extract has passed through the centrifuge, wash the feed mechanism (with centrifuge still running) with several increments of clean solvent, allowing each increment to run through the centrifuge until the effluent is essentially colorless.
3. 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 controlled at 230 6 9°F (110 6 5°C). Cool the container and redetermine the mass immediately. The increase in mass is the mass of mineral matter, W4, (12.1) in the extract.

Volumetric Method:

1. Place the extract in a previously tared and calibrated flask. Place the flask in a controlled-temperature bath controlled to 0.2°F (60.1°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 which has been kept at the same temperature. Bring the level of the liquid 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.
2. Calculate the volume of asphalt and fines in the extract as follows:

$$V_1 = V_2 - \frac{(M_1 - M_2)}{G_1}$$

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, and
G_1 = specific gravity of the solvent determined to the nearest 0.001.

3. Calculate the mass of fines in the extract as follows:

$$M_3 = K(M_2 - G_3V_1)$$

Where:
M_3 = mass of fines in the extract,
G_2 = specific gravity of fines.
G_3 = specific gravity of asphalts.

$K = G_2 \big/ G_2 - G_3$

Calculate the percent bitumen in the test portion as follows:

$$Bitumen\ content, \% = \left[\frac{(W_1 - W_2) - (W_3 + W_4)}{W_1 - W_2} \right] * 100$$

Where:

W 1 = mass of test portion,

W2 = mass of water in the test portion,

W3 = mass of the extracted mineral aggregate, and

W4 = mass of the mineral matter in the extract.

<div align="center">

Experiment No. 9
Standard Test Method For
EFFECT OF HEAT AND AIR ON ASPHALT IC MATERIALS (Thin-Film Oven Test)

</div>

Purpose: This test method covers the determination of the effect of heat and air on a film of semisolid asphalt materials. The effects of this treatment are determined from measurements of selected asphalt properties before and after the test.

Summary of Method:

- A film of asphalt material is heated in an oven for 5 h at 325 °F (163 °C). The effects of heat and air are determined from changes incurred in physical properties measured before and after the oven treatment.
- Precision values for the method have been developed for viscosity, viscosity change, penetration change, mass change, and ductility.

Standard Reference: ASTM D 1754-87

Apparatus Required:

- *Oven* - The oven shall be electrically heated for operating temperatures up to 356 °F (180 °C). The oven shall be rectangular with minimum interior dimensions of 13 in. in each direction. The oven shall have, in front, a tightly fitted hinged door, which shall provide a clear opening substantially the same as the interior height and width of the oven.
- *Rotating Shelf* - The oven shall be provided with a metal circular shelf having a minimum diameter of 9.8 in. The shelf construction shall be such that it provides a flat surface for support of the containers without blocking all air circulation through the shelf when the containers are in place.
- *Thermometer* - An ASTM Loss on Heat Thermometer having a range from 155 to 170 °C.
- *Container* - A cylindrical pan, 5½ in. in inside diameter and 3/8 in. deep with a flat bottom. Fifty milliliters of the sample in this size container give a film thickness of approximately 1/3 in. Pans shall be made of aluminum or stainless steel.

Preparation of Samples:

1. Place sufficient material for the test in a suitable container and heat to a fluid condition. Extreme care should be taken so that there is no local overheating of the sample and that the highest temperature reached is not more than 150 °C. Stir the sample with a general-purpose thermometer during the heating period, but avoid incorporating air bubbles in the sample. Weigh 50 ± 0.5 g into each of two or more tare containers.
2. At the same time, pour a portion of the sample into the containers specified for measurement of original asphalt properties. Complete the tests by appropriate ASTM test methods.
3. If the quantitative value of the loss is desired, cool the samples for the oven test to room temperature and weigh each sample separately to the nearest 0.001 g. If the

loss is not required, allow the samples to cool to approximately room temperature before placing in the oven.

Procedure:

1. Level the oven so that the shelf rotates in a horizontal plane. Determine the temperature of the oven by means of the specified thermometer.
2. With the oven at 325 ± 2 °F (163 ± 1 °C), quickly place the containers with the sample on the circular shelf, close the oven, and start rotating the shelf. Maintain the specified temperature range for 5 h after the sample has been introduced and the oven has again reached that temperature. The 5-h period shall start when the temperature reaches 162 °C and in no case shall the total time that a sample is in the oven be more than 5 1/4 h. At the conclusion of the heating period, remove the samples from the oven. If the loss is being determined, cool to room temperature, weigh to the nearest 0.001 g, and calculate the loss based on the asphalt in each container. If the loss is not being determined skip step No. 3, given below.
3. After weighing the samples, place them on a refectory-board and then on the shelf of the oven maintained at 163°C. Close the oven and rotate the shelf for 15 min, remove the samples and board.
4. Transfer the material from each pan immediately into an 8-oz ointment tin. Remove all of the material from the pans by scraping with a suitable spatula or putty knife. Stir the combined residues thoroughly, placing the 8-oz container on a hot plate to maintain the material in a fluid condition if necessary. Complete the tests on residue by appropriate ASTM test methods within 72 h of performing this test.

Experimental Data and Results:

- Tabulate the data in the given *Test Data Sheet*. Report the values of the original asphalt properties measured and the residue property values as measured. Viscosity change may also be expressed as the ratio of the residual asphalt viscosity to the original asphalt viscosity. Penetration change is evaluated as the penetration of the residue expressed as the percentage of the original penetration.
- Report ductility or other test results in accordance with the appropriate ASTM test methods.

Discussion: Discuss about the test and results in the given *Test Data Sheet*.

Experiment No. 9
Test Data Sheet
EFFECT OF HEAT AND AIR ON ASPHALTIC MATERIALS (Thin-Film Oven Test)

Sample No. _____ Description of Material: _____

Tested by: _____ Date of Testing: _____

Test Temperature: _____ °C; Type of Asphalt: _____;

Test No.	Loss Properties			Change in Asphalt Properties					
	Wt. of sample before test, g	Wt. of sample after test, g	% Loss	Original viscosity cst	Residue viscosity cst	Original pene-tration	Resi-due penet ration	Orig. Duc-tility	Resi due Duc-tility
1									
2									
3									
Average									

Discussion of Test Results: _____

_____

_____

_____

_____

Experiment No. 9
Standard Method of Test for
VISCOSITY DETERMINATIONS OF UNFILLED ASPHALTS USING THE
BROOKFIELD THERMOSEL APPARATUS

Objective:

Significance and Use:

Relevant Theory:

Standard Reference:

Apparatus Required:

Procedure Molding Test Specimen:

Testing:

Experimental Data and Results:

Discussion: