Designation: D 6847 – 02

Standard Test Method for Quantitative Extraction and Recovery of Asphalt Binder from Asphalt Mixtures[1]

This provisional standard is issued under the fixed designation D 6847; the number immediately following the designation indicates the year of original adoption.

1. Scope

1.1 This test method covers a procedure for the extraction and recovery of asphalt binder from asphalt mixtures—both HMA (hot mix asphalt) and RAP (reclaimed/recycled asphalt pavement)—having a minimal effect on the physical properties of the asphalt binder recovered. It is primarily intended for use when the physical properties of the recovered asphalt are to be determined. It can also be used to determine the quantity of asphalt binder in the HMA or RAP. Recovered aggregate may be used for sieve analysis. This may also be accomplished through Test Methods D 2172.

1.2 The values stated in SI units are regarded as the standard. Values in parentheses are for informational use.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—It is suggested that agency and personnel performing this test meet the requirements of Specification D 3666.

2. Referenced Documents

2.1 *ASTM Standards:*

D 75 Practice for Sampling Aggregates[2]

D 979 Practice for Sampling Bituminous Paving Mixtures[2]

D 1461 Test Method for Moisture or Volatile Distillates in Bituminous Paving Mixtures[2]

D 2172 Test Methods for Quantitative Extraction of Bitumen From Bituminous Paving Mixtures[2]

D 2939 Test Methods for Emulsified Bitumens Used as Protective Coatings[3]

D 3666 Specification for Minimum Requirements for Agencies Testing and Inspecting Road and Paving Materials[2]

D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Testing Soil, Rock, and Related Construction Materials[4]

D 5361 Practice for Sampling Compacted Bituminous Mixtures for Laboratory Testing[2]

D 5444 Test Method for Mechanical Size Analysis of Extracted Aggregate[2]

E 11 Specification for Wire Cloth and Sieves for Testing Purposes[5]

3. Summary of Test Method

3.1 The asphalt mixture is repeatedly washed and filtered with solvent in an extraction/filtration apparatus. Each filtrate is distilled under vacuum in a rotary evaporator with the asphalt remaining in the flask. After recovery of the final filtrate, the solution is concentrated to about 300 mL and centrifuged to remove aggregate fines. The decanted solution is distilled under vacuum to remove the extraction solvents. Nitrogen gas is introduced during the final phase of distillation to drive off any remaining traces of solvents. The quantity of asphalt binder in the asphalt mixture is calculated (optional) and the recovered asphalt (distillation residue) sample is subjected to further physical testing as required. The recovered aggregate can then be used for sieve analysis, if desired.

4. Significance and Use

4.1 This standard is used to extract and recover asphalt binder from an asphalt mixture sample. The recovered mixture components (asphalt binder and aggregates) can then be subjected to further physical tests.

5. Apparatus

5.1 *Extraction Vessel*—The extraction vessel shall be a device as shown in Fig. 1, and shall have a 130-mm long piece of 150-mm I.D. Schedule 80 Aluminum or Schedule 80, Grade 304 stainless steel pipe (see Fig. 2), with removable top and bottom plates (13-mm thickness). The top plate (see Fig. 3) shall have a mixing motor mount and 19-mm port for adding solvent. The bottom plate (see Fig. 4) shall be equipped with a quick connect fitting. Four 100-mm by 25-mm baffles (see Fig. 5) shall be mounted in the extraction vessel along with the following inserts in order as shown in Fig. 1: a 3-mm aluminum or stainless steel ring, sieve cloth, 2.00 mm (No.10)

[1] This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.25 on Analysis of Bituminous Mixtures.

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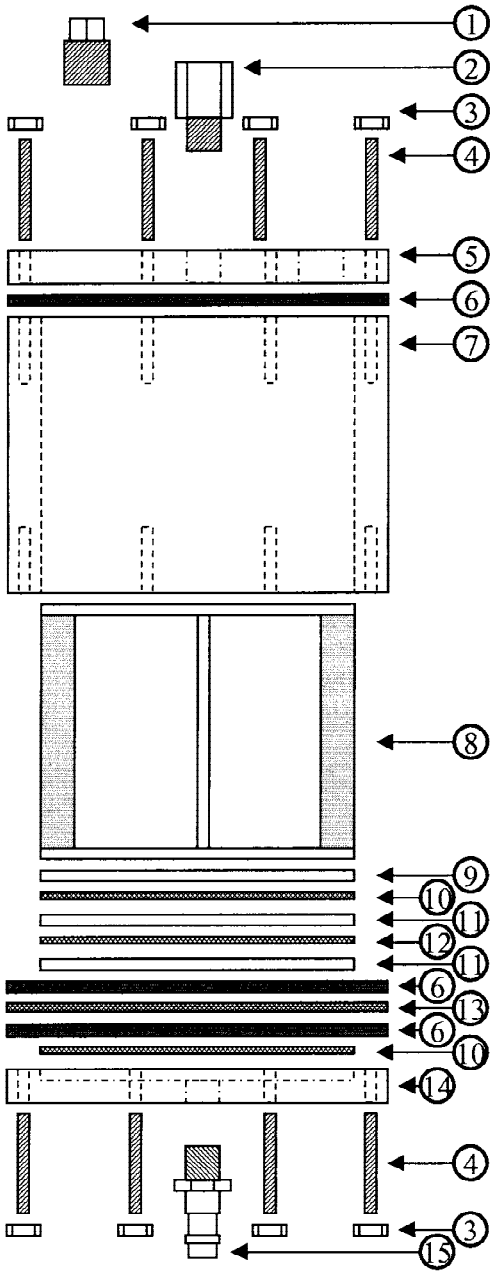
[2] *Annual Book of ASTM Standards*, Vol 04.03.

[3] *Annual Book of ASTM Standards*, Vol 04.04.

[4] *Annual Book of ASTM Standards*, Vol 04.08.

[5] *Annual Book of ASTM Standards*, Vol 14.02.



1- 1- ISO 12RT (3/4 in. NPT) plug
2- ISO 8RT (1/2 in. NPT) fitting with I.D. hole for motor shaft
3- 12 M5 x 0.5 nuts
4- 12 M5 x 0.5 x 50 mm studs
5- extraction vessel top
6- 3 Viton gaskets 3 mm thick, 16 mm width with holes to fit over studs
7- extraction vessel housing
8- baffle
9- metal ring 3-mm thick 148-mm O.D., 10 mm width
10- stainless steel #10 screen
11- metal spacers
12- # 50 screen
13- # 200 screen
14- extraction vessel bottom
15- ISO 4RT (1/4 in. NPT) quick connect fitting

FIG. 1 Extraction Vessel

openings;spacer (see Fig. 6); sieve cloth, 300mm (No. 50) openings; another spacer (see Fig. 6); a 3-mm gasket; sieve cloth, 75 mm (No. 200) openings; another 3-mm gasket; another sieve cloth, 2.00 mm (No. 10) openings.

NOTE 2—All references to stainless steel materials should be Grade 304 stainless steel to assure corrosion resistance. Sieve cloth should meet Specification E 11 specifications. Brass sieve cloth is acceptable, however, brass will require replacement more often due to wear from aggregate.

NOTE 3—If aluminum is used for the vessel several blank tests should be run to condition the aluminum. After this conditioning the vessel should never be polished to a bright shiny finish. If vessel is polished, recondition before running any tests.

5.2 *In-line Filter*—The in-line filter apparatus shall be a cartridge type with 20-mm retention and at least 820-cm^3 effective filter area. The filter apparatus shall be removable to accommodate weighing before and after the procedure. The filter shall be capable of withstanding heat up to 135°C (275°F), without degradation in order to accommodate oven drying of the filter apparatus.

NOTE 4—Whatman Polycap 75 HD catalog number 6703-7521 or equivalent is a suitable filter.

5.3 *Filtrate Vacuum Flasks*—Two (2) filtrate vacuum flasks, 1000 mL.

5.4 *Round Bottom Flask*—Round Bottom recovery flask (1000 mL).

5.5 *Flowmeter*—Gas flowmeter, capable of indicating a gas flow up to 1000 mL/min.

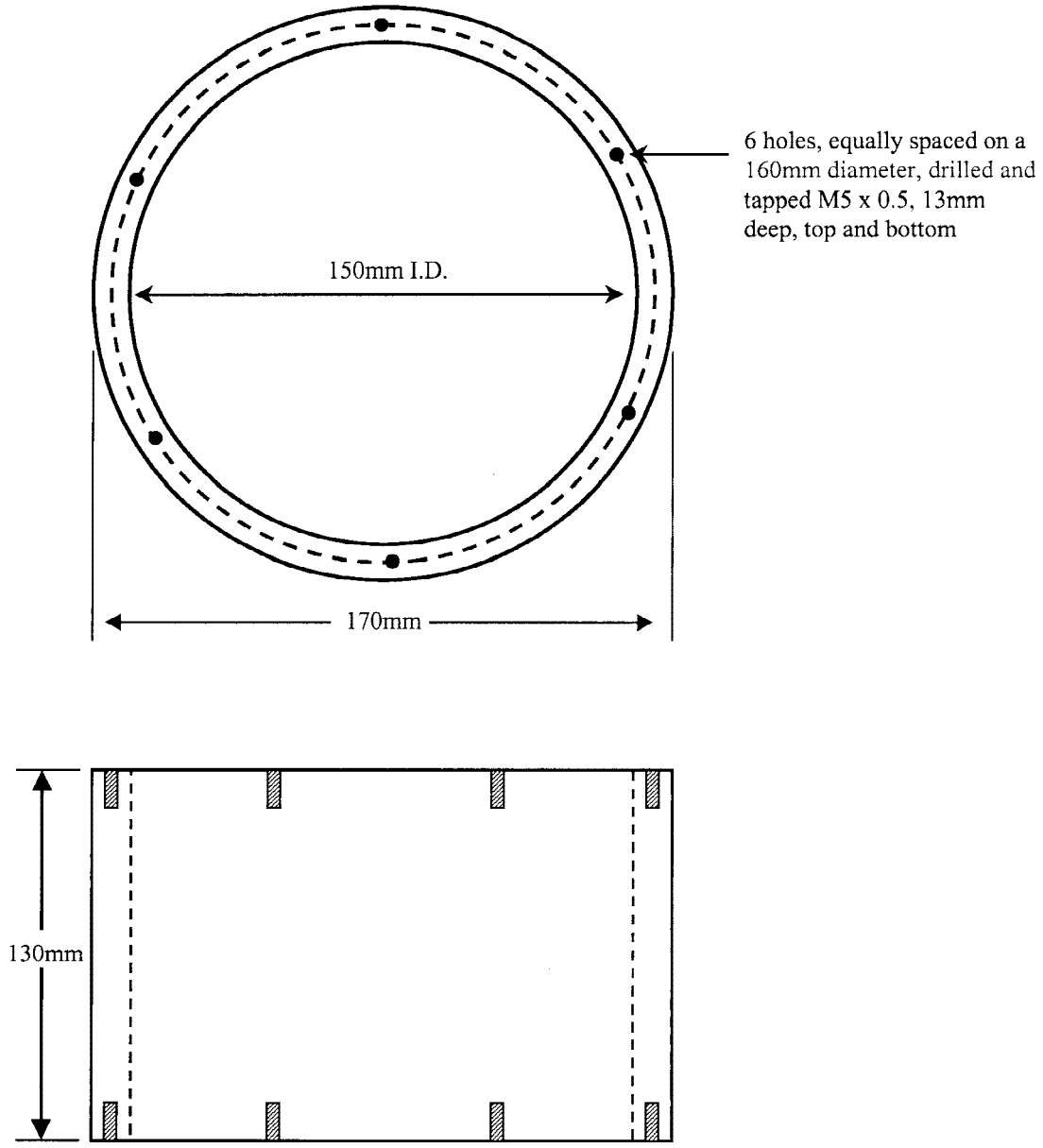


FIG. 2 Extraction Vessel Housing

5.6 *Rotary Evaporator*—Rotary Evaporator device, with transfer and purge tubes, capable of holding a recovery flask in oil at a 15° angle and rotating at 40 RPM.

NOTE 5—The Buchi Rotavapor R-200 or equivalent has proven acceptable for these requirements.

5.7 *Oil Bath*—Hot oil bath, capable of heating oil to 180°C (356°F).

5.8 *Mixing Motor*—Single speed mixing motor, 150 W (1/5 hp), 30 RPM.

5.9 *Centrifuge*—Batch unit capable of exerting a minimum centrifugal force of 770 times gravity.

5.10 *Centrifuge Bottles*—Wide mouth centrifuge bottles, 250 mL.

5.11 *Oven*—Oven capable of maintaining a temperature of 110 ± 5°C (230 ± 10°F).

5.12 *Balance*—Balance of suitable capacity meeting the requirements of Specification D 4753, class GP2.

5.13 *Thermometer*—Thermometer, including a range of 100 to 200°C (212 to 390°F) with subdivisions of 2°C (5°F).

5.14 *Utilities*—Vacuum source and cooling water source.

NOTE 6—The vacuum pump should be able to achieve absolute pressure of 8.0 kPa (2.4 in Hg absolute).

5.15 *Boiling Beads*—3-mm glass boiling beads.

6. Reagents and Materials

6.1 *6-mm Diameter Polypropylene Tubing*, varying lengths, for transferring solution throughout the apparatus.

NOTE 7—To avoid contamination of the sample due to solvent degradation of the tubing, do not substitute Nalgene or rubber tubing for the polypropylene tubing specified.

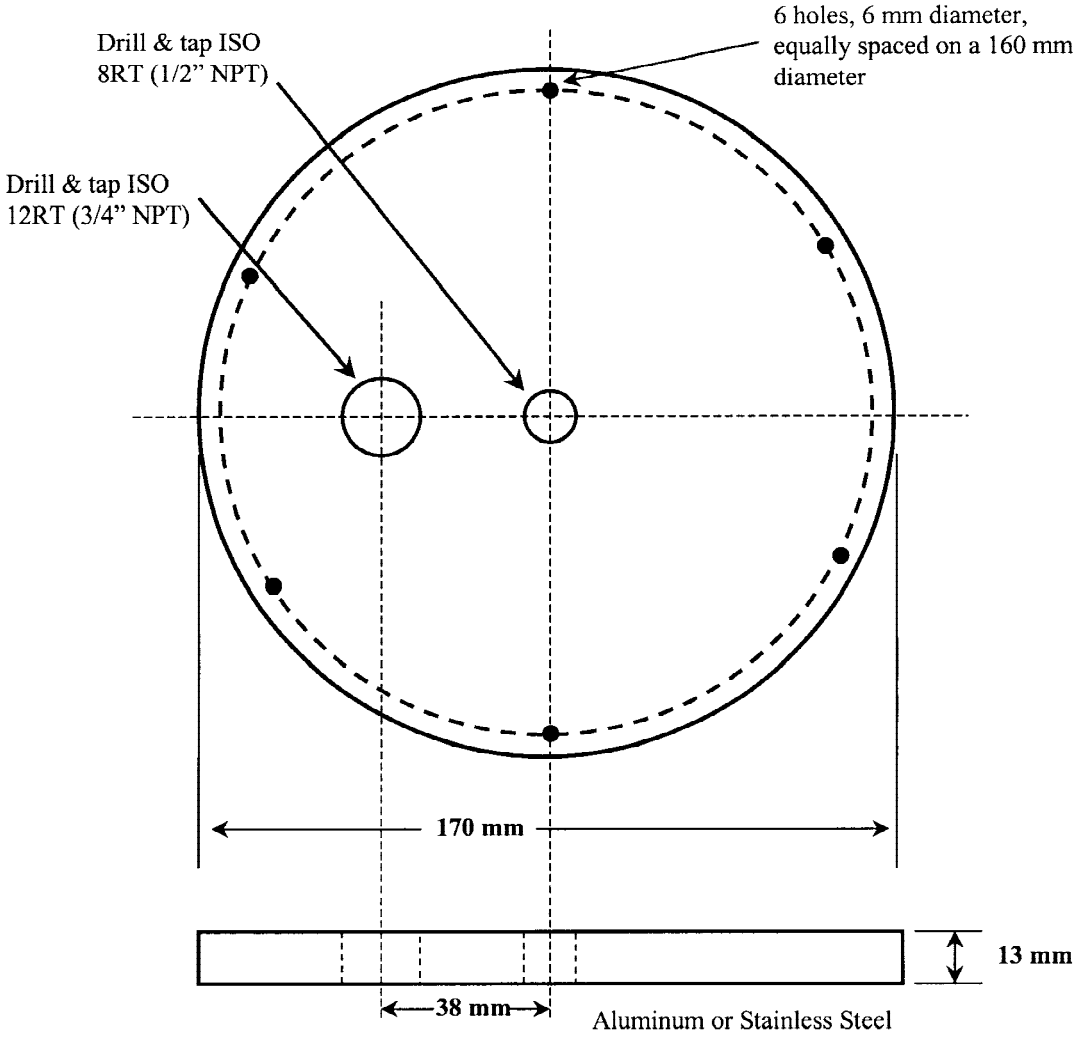


FIG. 3 Extraction Vessel Top Plate

6.2 *Copper Tubing*, of an amount and size adequate to connect the apparatus as shown in Fig. 7.

NOTE 8—New copper tubing should be conditioned by running several blank tests before use for regular testing. Stainless steel tubing or any other tubing not affected by solvents may be used.

6.3 *Solvent*:

6.3.1 *n*-Propyl Bromide, or

6.3.2 Trichloroethlylene (reagent grade), or,

6.3.3 Toluene (reagent grade). If using Toluene, combine with Ethanol (absolute) in proportions of 85 % Toluene and 15 % Ethanol after the third wash (in 11.2.6).

6.4 *Nitrogen Gas*, at least 99.95 % pure, in a pressurized tank, with a pressure-reducing regulator valve.

7. Hazards

7.1 Use solvents only under a fume hood or with an effective surface exhaust system in a well-ventilated area and observe the manufacturer's recommended safety precautions when using compressed nitrogen.

8. Sampling, Test Specimens, and Test Units

8.1 Obtain asphalt mixture samples in accordance with Practice D 979. When sampling from a compacted roadway, remove specimens from the roadway in accordance with Practice D 5361. When sampling RAP, refer to Practice D 75 for aggregate sampling.

9. Preparation of Apparatus

9.1 *Preparing the Extraction Vessel*—Install the baffle piece and other internal parts in the order as shown in Fig. 1. Tightly and evenly fasten the bottom (with the quick connect fitting in-place) of the vessel with wing nuts or hexagonal nuts.

9.2 *Preparing the Rotary Evaporator*—Turn on the cooling water. Turn on the oil bath and set the temperature to 100 ± 2.5°C (212 ± 5°F). Place six boiling beads in the 1000 mL round bottom recovery flask. Attach this flask to the rotary evaporator. During the procedure the flask will be immersed approximately 38 mm into the oil bath. Set the angle of the recovery flask at 15° from the horizontal of the bath. Set the flask rotation at 40 RPM. Clamp the empty condensate flask

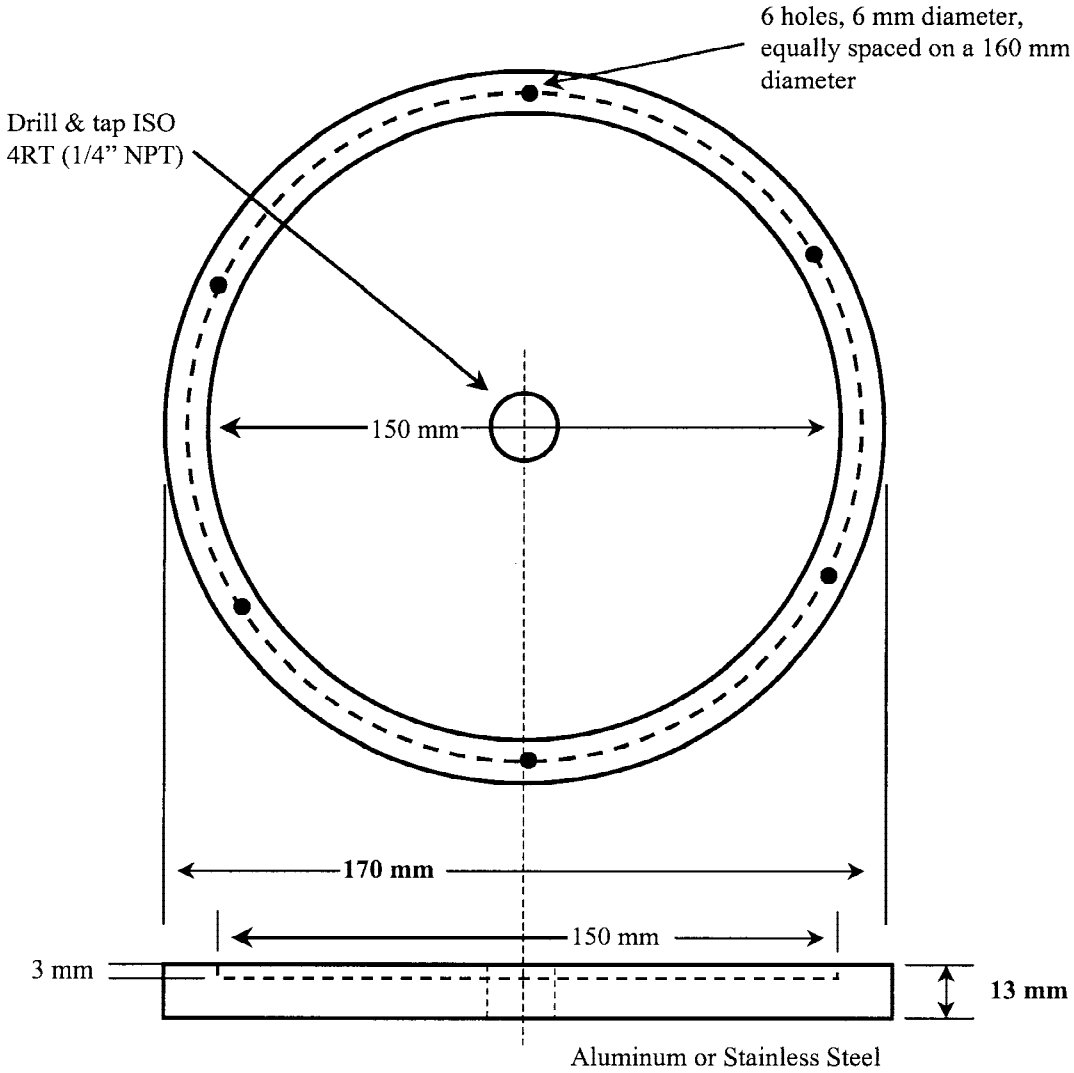


FIG. 4 Extraction Vessel Bottom Plate

onto the condenser. Attach the transfer tube inside the neck of the rotary evaporator. Attach the filtrate transfer line to the external fitting on the neck of the rotary evaporator.

10. Calibration and Standardization

10.1 At least every six months, verify the calibration of the oil bath temperature indicator by using a calibrated mercury in glass thermometer of suitable range that is accurate to ± 1°C. Immerse the thermometer in the oil bath and compare the temperature indicated by the calibrated thermometer to the temperature display on the oil bath.

10.2 At least every six months, use residual pressure manometer or calibrated absolute pressure gage to verify calibration of the vacuum indicator.

10.3 At least every six months, verify the rotational velocity of the rotary evaporator.

10.4 At least every six months, verify the flow rate of the nitrogen flow meter.

11. Procedure

11.1 *Sample Preparation*:

11.1.1 If a test sample of asphalt mixture is not sufficiently soft to separate with a spatula or trowel, place the test sample in a large, flat pan and warm it in an oven at 110 ± 5°C (230 ± 9°F) only until it can be easily handled with gloves or mixed.

11.1.2 Split or quarter the loose asphalt mixture until an amount of the test sample that will yield approximately 50 to 60 g of recovered asphalt binder is obtained (Approximately 1,000 g of original asphalt mixture).

NOTE 9—This procedure works best when recovering less than 60 g of asphalt binder at one time. If the asphalt binder content of the mix is already known, then the mass of the asphalt mixture required is that which would yield 50 to 60 g of asphalt binder.

NOTE 10—The maximum aggregate size in the test specimen will affect the calculated asphalt content. If the calculated results from this standard are used to represent the asphalt content in the asphalt mixture from which the sample was obtained, use a sample mass that will ensure that inclusion or removal of one maximum size particle will not change the calculated asphalt content by more than 0.05 %. This may require testing multiple specimens.

11.1.3 If the determination of the asphalt binder content is required, refer to the minimum sample size referenced in Test

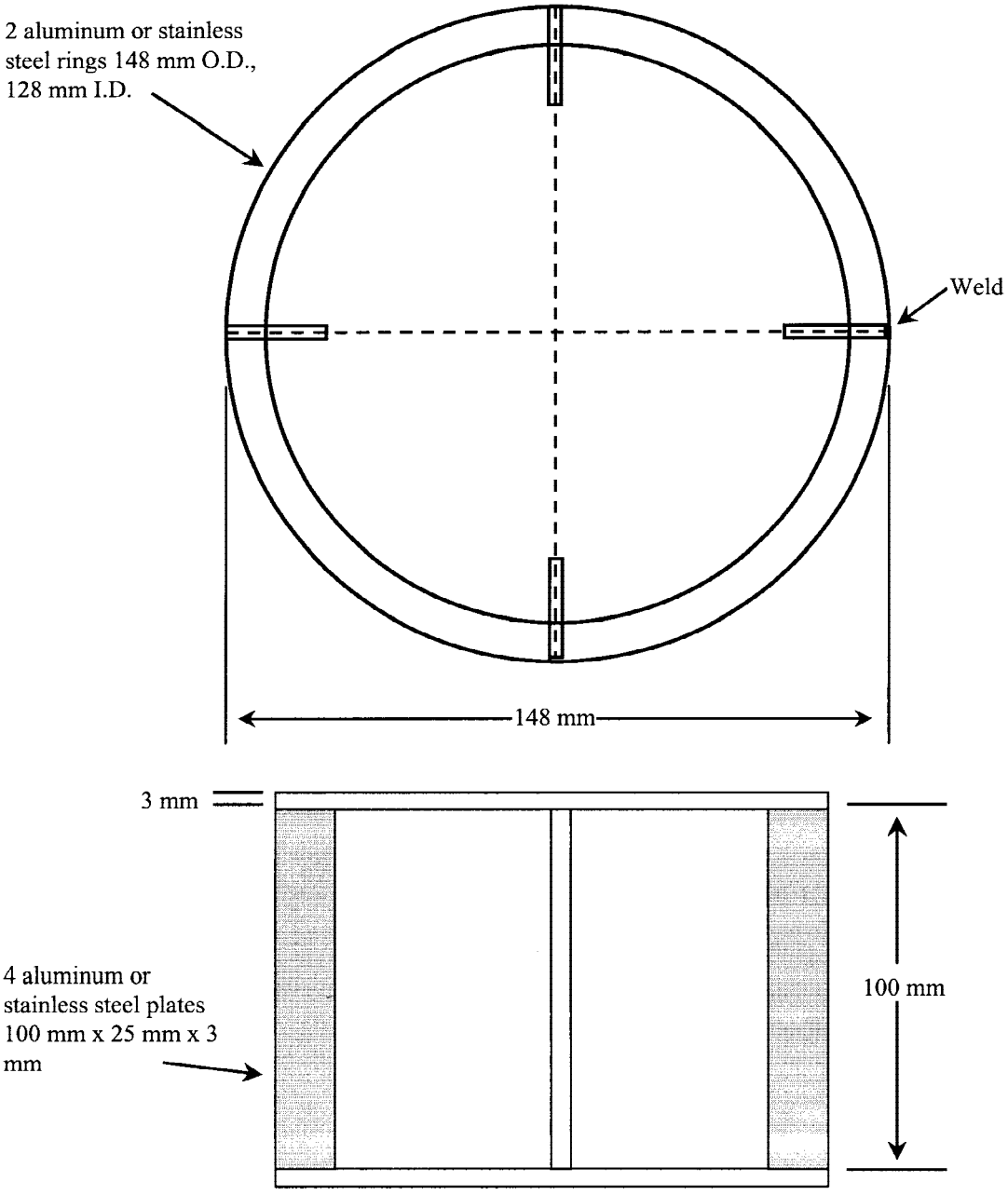


FIG. 5 Extraction Vessel Baffle

Method D 2172. If the determination of gradation is required, refer to Test Method D 5444.

11.2 *Extraction and Filtration*:

11.2.1 Place the test sample in the extraction vessel. Put the gasket and the upstream end on the vessel and fasten the nuts tightly and evenly, ensuring a secure seal.

11.2.2 Pour 600 mL of solvent through the 19-mm port on the upstream end of the extraction vessel. Blanket the interior of the vessel by injecting nitrogen through this same port at a rate of 1000 mL/min for one min. Close the port with the threaded plug. Attach the vessel to the motor and rotate the vessel for 5 ± 1 min at 30 RPM. Turn off motor.

11.2.3 Remove the extraction vessel, place it on a stand and attach the quick connect fitting to the vessel such that the effluent will flow to the first filtrate receiving flask. Make sure the first filtrate flask's transfer line is closed. Remove the port plug from the upstream end of the vessel and blanket the extractor with nitrogen at a rate of 400 mL/min while drawing the asphalt/solvent solution into the first flask. Apply enough vacuum to the first filtrate receiving flask to draw the material from the vessel. Continue drawing the solution into the first flask until there is no noticeable amount of solution exiting the vessel. Turn off the vacuum.

11.2.4 To filter through the in-line cartridge filter, switch the vacuum to the second filtrate-receiving flask. Filter until there is no noticeable amount of solution remaining in the first flask or the filter. Turn off the vacuum.

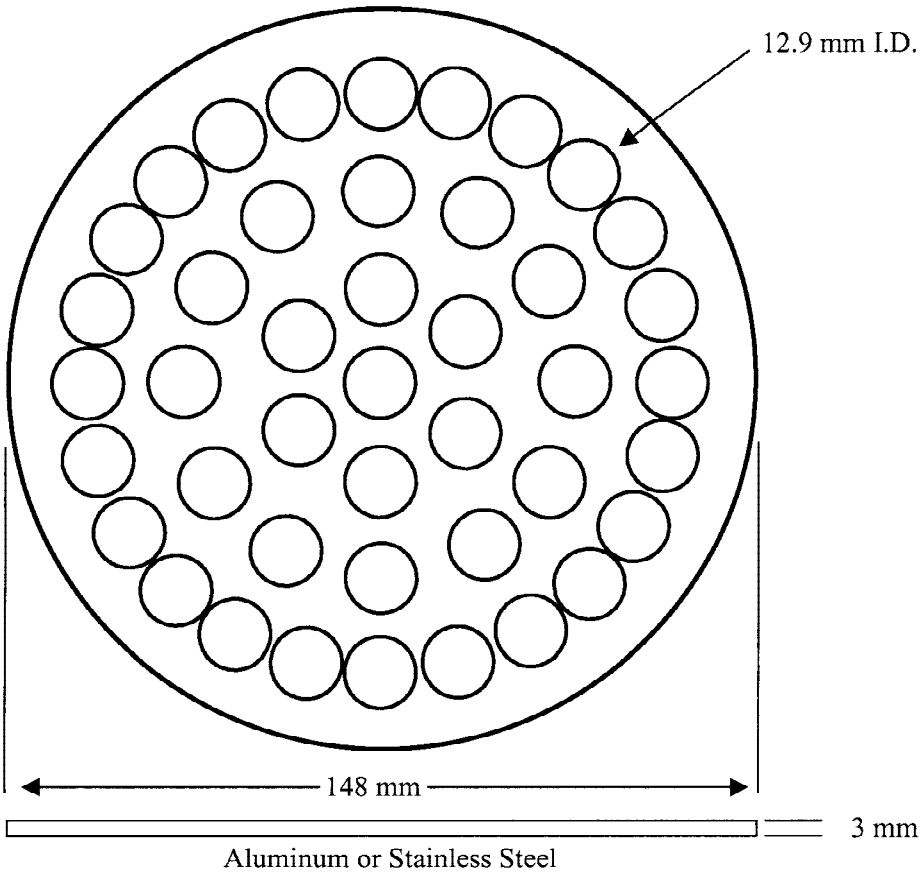


FIG. 6 Extraction Vessel Spacer

11.2.5 After filtration, open the filtrate transfer valve on the second receiving flask and allow the solution to flow into the recovery flask. Continue the transfer until the second filtrate receiving flask is empty or the recovery flask is about ⅔full. At this point, begin primary distillation (see 11.3).

11.2.6 After the distillation is started, disconnect the extraction vessel from the quick connect fitting and repeat the extraction procedure (see 11.2.2-11.2.5). For this second wash, use 400 mL of solvent and mix/rotate for 10 ± 1 min. For all subsequent washes, use 400 mL of solvent and mix/rotate for 30 ± 1 min.

NOTE 11—After the third wash, the condensate from the primary distillation step may be used for the extraction solvent. Recycling solvent in this manner allows the entire procedure to use approximately 1500 mL solvent.

11.2.7 Proceed to the final recovery step (see 11.4) when the filtrate flowing through the transfer tubes is a "light straw" color. A minimum of three washes is required.

11.3 *Primary Distillation*:

11.3.1 Close the filtrate transfer valve line to the rotary evaporator and distill the solvent at 100 ± 2.5°C (212 ± 5°F) oil bath temperature and 21.3 ± 3.3 kPa (6.6 ± 1.0 in Hg) absolute pressure.vacuum.

11.3.2 If, after the primary distillation step the condensate flask is over half full, pour off the solvent from the flask. Save this solvent for use in subsequent washes (see Note 11). After primary distillation of each filtrate, maintain vacuum, temperature, flask rotation, and cooling water. Repeat the primary distillation after each filtration (see Note 12).

NOTE 12—It is important to concentrate the asphalt in the recovery flask after each wash. This reduces the time the binder is in the solvent and, therefore, minimizes asphalt hardening.

11.4 *Final Extraction and Recovery*:

11.4.1 Distill the contents of the recovery flask until it is about ⅓ full.

11.4.2 Turn off vacuum, then clean and disconnect the recovery flask before pouring the contents into the centrifuge bottles using a funnel and screen to prevent the boiling beads from entering the bottles. Fill the bottles, in their respective holders, so that their masses are equal. Wash any remaining residue from the recovery flask into the centrifuge bottles. Additional solvent may be necessary to make the masses of the bottles equal. Spin the bottles in the centrifuge at minimum of 770 times gravity for 25 min.

11.4.2.1 While centrifuging the material, increase the oil bath temperature to 174 ± 2.5°C (345 ± 5°F).

11.4.3 Empty the liquid content of the centrifuge bottles back into the recovery flask and add the six 3-mm diameter glass boiling beads. Reattach the flask to the rotary evaporator. Disconnect the filtrate transfer line from the external rotary evaporator neck fitting and replace it with the nitrogen gas line. Apply 21.3 ± 3.3 kPa (6.6 ± 1.0 in Hg) absolute pressure. Lower the flask approximately 38 mm into the oil bath.

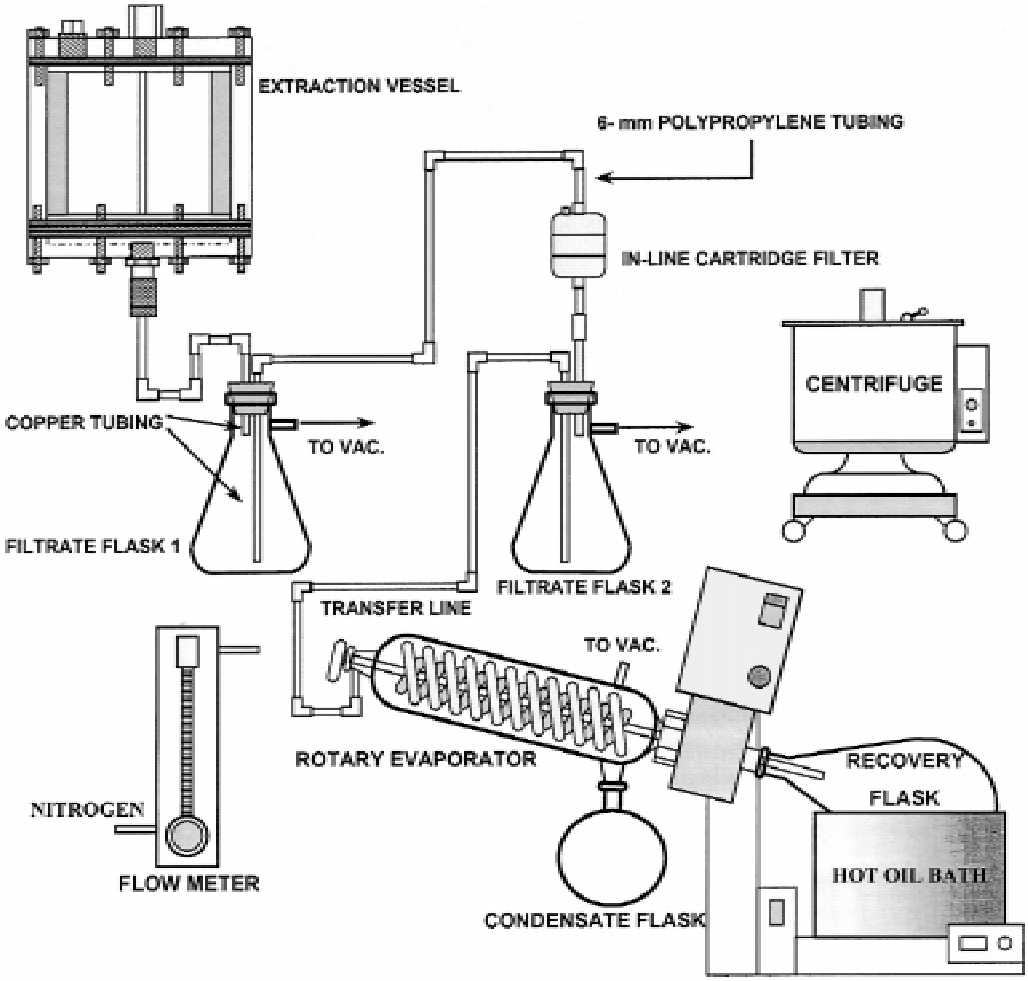


FIG. 7 Extraction and Recovery Apparatus

11.4.4 Distill the solvent.

11.4.5 When the condensation rate falls below one drop every 30 s, introduce the nitrogen gas at a rate of 1000 mL/min. Maintain the gas flow, vacuum and bath temperature for 30 ± 1 min to reduce the residual solvent concentration to near zero. Complete removal of residual solvent is very important for obtaining accurate asphalt properties.

11.4.6 Shut down the oil bath, flask rotation, vacuum, gas flow, and cooling water. Remove the recovery flask. Pour the asphalt into a sample tin using a screen to prevent the boiling beads from entering the tin.

NOTE 13—It is suggested that ash content should be determined according to Test Methods D 2939 when using unfamiliar aggregate and annually to assure the extraction equipment is working properly. Ash content should be less than 1 % by weight of asphalt binder.

12. Calculation or Interpretation of Results

12.1 When a determination of asphalt binder content is desired, use the following procedure:

Before section 11.2.1:
Determine mass of test sample, $M_{org\ mix}$ Determine mass of cartridge filter Determine mass of centrifuge bottles

After section 11.4.3:
Dry centrifuge bottles, cartridge filter, and opened vessel (including inserts) in a 110°C oven to constant mass Determine mass of fine material in centrifuge bottles (dry − tare), ΔBottles Determine mass of fine material in filter (dry − tare), ΔFilter Determine mass of all recovered aggregate material in vessel (scrape/brush all cloths, etc.), M_{RA} Asphalt Content % = $[M_{org\ mix} - (M_{RA} + \Delta Bottles + \Delta Filter)]/M_{org\ mix}$

13. Report

13.1 Report source of test sample.

13.2 Report the following, if the asphalt binder content is to be determined:

13.2.1 The mass of test sample to the nearest gram,

13.2.2 The mass of asphalt binder in the test sample to the nearest gram, and

13.2.3 The percent asphalt binder in the test sample to the nearest 0.01 %.

14. Precision and Bias

14.1 *Precision*—The multi-lab precision of the procedure for measuring asphalt binder content is being determined and will be available on or before August 2006. It is not feasible to specify the multi-lab precision of the procedure at this time because there are currently only a few laboratories equipped to run this method. Therefore, this test method should not be used for acceptance or rejection of a material for purchasing purposes.

Test and Type Index	Standard Deviation, %, (1s)	Acceptable Range of Two Test Results, %, (d2s)
Single-operator precision	0.18	0.51

14.2 *Bias*—No information can be presented on the bias of the procedure for measuring asphalt binder content because no material having an accepted reference value is available.

15. Keywords

15.1 asphalt binder; extraction; recovery; rotary evaporator