

**Standard Test Method for the
Quantitative Extraction and Recovery of
Asphalt Binder from Asphalt Mixtures**

AASHTO Designation: TP2-01^{1, 2}

1. Scope

1.1 This standard describes a procedure for the extraction and recovery of asphalt binder from asphalt mixtures [both hot mix asphalt (HMA) and reclaimed asphalt pavement (RAP)] which has a minimal effect on the physical and chemical properties of the asphalt binder recovered. It is intended for use when the physical or chemical properties or both of the recovered asphalt binder 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.

1.2 This method is applicable to HMA sampled from the pavement, RAP sampled from the pavement or stockpile, HMA plant production, or laboratory fabricated HMA.

1.3 This procedure may involve hazardous materials, operations and equipment. This procedure does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 AASHTO Standards

M231	Weighing Devices Used in the Testing of Materials
T110	Moisture or Volatile Distillates in Bituminous Paving Mixtures
T168	Sampling Bituminous Paving Mixtures

2.2 ASTM Standards

D75	Sampling Aggregates
D5361	Sampling Compacted Bituminous Mixtures for Laboratory Testing

3. Terminology

3.1 Asphalt binder - an asphalt-based cement that is produced from petroleum residue either with or without the addition of non-particulate organic modifiers

4. **Summary of Method** - 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 and chemical testing as required. The recovered aggregate can then be used for sieve analysis, if desired.

5. **Significance and Use** - This method is used for obtaining recovered asphalt binder residue samples from asphalt mixture samples for further physical and chemical analyses, and for the optional calculation of asphalt binder content.

6. Apparatus

6.1 Extraction Vessel - The extraction vessel shall be a device as shown in Figure 1, and shall have a 130-

¹ This standard is based on SHRP Product 1004.

² Approved in October 1994, this provisional standard was first published in March 1995. It was reapproved in 1996. The 2001 publication includes major changes resulting from NCHRP 9-12 project.

mm long piece of 150-mm I.D. Schedule 80 aluminum pipe or Schedule 80, grade 304 stainless steel pipe (Figure 2) with removable top and bottom 13-mm thick aluminum or stainless steel plates. The top plate (Figure 3) shall have a mixing motor mount and 19-mm port for adding solvent. The bottom plate (Figure 4) shall be equipped with a quick connect fitting. Four 100-mm by 25-mm baffles (Figure 5) shall be mounted in the extraction vessel followed by a 3-mm aluminum ring, 2-mm (#10) mesh screen, spacer (Figure 6), 0.3-mm (#50) mesh screen, another spacer, 0.075-mm (#200) mesh screen, then another 2-mm (#10) mesh screen, as shown in Figure 1.

Note 1 - Vessel available through Pass Industries, telephone number (606)881-0205, has proven acceptable for these requirements.

6.2 In-line filter - The in-line filter apparatus shall be a cartridge type with 20- μ m retention and at least 820cm³ effective filter area. The filter apparatus shall be able to be removed from the system to accommodate weighing before and after procedure. The filter shall be capable of withstanding heat up to 135°C without degradation in order to accommodate oven drying of the filter apparatus.

Note 2 - Whatman Polycap™ 75 HD Catalog number 6703-7521 or equivalent is a suitable filter.

6.3 Two (2) filtrate flasks with tubulation, 1000mL.

6.4 Round bottom flasks, 1000 mL and cork stands

6.5 Gas flowmeter, capable of indicating a gas flow up to 1000 mL/minute

6.6 Rotary evaporator device, with transfer and purge tubes, capable of holding a recovery flask in oil at a 15 degree angle and rotating at 40rpm

Note 3 - The Buchi Rotavapor RE-120 has proven acceptable for these requirements.

6.7 Hot oil bath, capable of heating oil to 180°C

6.8 Single speed mixing motor, 150 W (1/5 hp), 30 rpm

6.9 Centrifuge, batch unit capable of exerting a minimum centrifugal force of 770 times gravity

6.10 Wide-mouth centrifuge bottles, 250 mL.

6.11 Oven, capable of maintaining a temperature of $110 \pm 5^\circ\text{C}$

6.12 Balance, of suitable capacity meeting the requirements of M231 for Class G2 balances

6.13 Thermometer, having a range of 30 to 300°C

6.14 Utilities - Vacuum source and cooling water source.

6.15 Scale (optional) - having a capacity of 12 kg or more, sensitive to 0.1 g or less, and accurate within 0.1% of the test load at any point within the range of use for this test. Within any 100-g range of test load, a difference between readings shall be accurate within 0.1 g.

7. Materials and Reagents

7.1 6-mm diameter polypropylene tubing of varying length, for transferring solution throughout the procedure

Note 4 - 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.

7.2 Copper tubing, of an amount and size adequate to connect the apparatus as shown in Figure 6 .

7.3 Solvent

7.3.1 n-Propyl Bromide, or

7.3.2 Trichloroethylene, reagent grade, or

7.3.3 Toluene, reagent grade. If using Toluene, combine with Ethanol, absolute, in proportions of 85% Toluene and 15% Ethanol after the third wash (in section 12.2)

7.4 Nitrogen gas, at least 99.95 percent pure, in a pressurized tank, with a pressure-reducing regulator valve

8. Hazards - 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.

9. Sampling - Obtain asphalt mixture samples in accordance with T168. When sampling from a compacted roadway, remove specimens from the roadway in accordance with ASTM D5361. When sampling RAP, refer to ASTM D75 for aggregate sampling.

10. Preparation of Apparatus

10.1 Preparing the extraction vessel - Install the baffles piece and other internal parts in the order shown in Figure 1. Tightly and evenly fasten the bottom piece (with quick connect fitting) of the vessel with wing nuts or hexagonal nuts.

10.2 Preparing the rotary evaporator - Turn on the cooling water. Turn on the oil bath and set the temperature to $100 \pm 2.5^{\circ}\text{C}$. Place six 3-mm glass boiling beads in a 1000 mL round bottom flask. Attach this recovery flask to the rotary evaporator and immerse approximately 38 mm of the flask into the oil bath. Set the angle of the recovery flask from the horizontal to the bath at 15 degrees. Set the flask rotation at 40 rpm. Clamp the empty condensate flask onto the condenser. Attach the transfer tube inside neck of rotary evaporator. Attach the filtrate transfer line to the external fitting on neck of rotary evaporator.

11. Standardization

11.1 At least every six months, verify the calibration of the oil bath temperature detector by using a certified mercury in glass thermometer of suitable range that is accurate to $\pm 0.2^{\circ}\text{C}$. Immerse the thermometer in the oil bath close to the thermal detector and compare the temperature indicated by the certified thermometer to the temperature setting for the oil bath. If the temperature indicated by the thermal detector does not agree with the certified thermometer within $\pm 0.5^{\circ}\text{C}$, perform additional calibration or maintenance.

11.2 At least every six months, use a mercury manometer or other certified pressure measurement device to verify calibration of the vacuum indicator. If the vacuum indicator and the certified pressure measurement device do not agree within ± 0.1 kPa, perform additional calibration or maintenance.

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

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

12. Procedure

12.1 Sample Preparation

12.1.1 If a sample of asphalt mixture is not sufficiently soft to separate with a spatula or trowel, place the sample in a large, flat pan and warm it in an oven at $110 \pm 5^{\circ}\text{C}$ only until it can be handled or mixed.

12.1.2 Split or quarter the loose asphalt mixture sample until an amount of the sample that will yield approximately 50 to 60 g of extracted asphalt binder is obtained typically approximately 1000g of asphalt mixture.

Note 5 - This procedure works best when recovering less than 60 g of asphalt binder. Therefore, if the asphalt binder content of the mix is already known, then the mass of the original sample required is that which yields about 50 to 60 g of asphalt binder.

Note 6 - 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 minimum mass of test specimens for calculations that will ensure that inclusion or removal of one maximum size particle will not change the calculated asphalt content by more than 0.05 percent. This may require testing multiple test specimens.

12.1.3 If the asphalt binder content is to be determined, obtain a separate test specimen from the asphalt mixture sample, determine the moisture content in accordance with T110 and record the mass percent of water in the test specimen.

12.2 Extraction and filtration

12.2.1 Place the asphalt mixture sample in the extraction vessel. Put the gasket and the upstream end piece on the vessel and fasten the wing nuts tightly and evenly, creating a secure seal.

12.2.2 Charge 600 mL of solvent through the 19-mm port on the upstream end of the extractor. Blanket the

interior of the extraction vessel by injecting nitrogen through the upstream port at a rate of 1000 mL/min for 1 minute. Close the port with the threaded plug. Attach the extractor to the motor. Start the motor and mix for 5 ± 1 minutes at 30 rpm. Turn off the motor.

12.2.3 Remove the extractor, place it on a stand and attach the quick connect fitting to the first filtrate receiving flask. Make sure the filtrate transfer line is closed. Remove the upstream port and blanket the extractor with nitrogen at a rate of 400 mL/min while drawing the asphalt/solvent solution into the first flask. Apply 93.3 ± 0.7 kPa (700 ± 5 mm Hg) 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.

12.2.4 Filtering through the in-line cartridge filter, switch the vacuum to the second filtrate receiving flask and apply 93.3 ± 0.7 kPa (700 ± 5 mm Hg) vacuum. Filter until there is no noticeable amount of solution remaining in the first flask or the filter. Turn off the vacuum.

12.2.5 After filtration, open the filtrate transfer valve on the second receiving flask and allow the solution to flow from the filtrate receiving flask to the recovery flask. Continue the transfer until the filtrate receiving flask is empty or the recovery flask is about 2/3 full, then, begin the primary distillation.

12.2.6 After the distillation is started, disconnect the extractor from the quick connect fitting. Repeat the extraction procedure. For the second wash use 400 ± 10 mL of solvent and mix/rotate for 10 ± 1 minutes. For all subsequent washes (Note 7), use 400 ± 10 mL of solvent and mix for 30 to 35 minutes.

Note 7 - After the third wash, the condensate from the primary distillation step be used for extraction solvent. Recycling solvent in this manner allows the entire procedure to use approximately 1500 mL solvent.

12.2.7 Proceed to the final recovery step (Section 12.4) when the filtrate flowing through the transfer tube, after a 30 minute wash, is a light brown color. A minimum of three washes is required.

12.3 Primary Distillation

12.3.1 Close the filtrate transfer valve line and distill solvent at $100 \pm 2.5^\circ\text{C}$ (oil bath temperature) and 93.3 ± 0.7 kPa (700 ± 5 mm Hg) vacuum.

12.3.2 If after the primary distillation step the condensate flask is over half full, empty the flask. Save this solvent for use in subsequent washes (Note 7). After primary distillation of each filtrate, maintain vacuum, temperature, flask rotation, and cooling water. Repeat the primary distillation after each filtration (Note 8).

Note 8 - It is important to concentrate the asphalt in the recovery flask after each wash and at a low temperature. This minimizes the temperature and the time spent in dilute solution and, therefore, minimizes asphalt hardening in solvent.

12.4 Final Extraction and Recovery

12.4.1 Distill the contents of the recovery flask until it is about 1/3 full.

12.4.2 Turn off the vacuum, then clean and disconnect the recovery flask and pour the contents into the centrifuge bottles using a funnel and screen to prevent the boiling beads from entering the bottles. Fill the bottles so that their masses are equal. Wash any remaining residue from the recovery flask into the centrifuge bottles. Increase the oil bath temperature to $174 \pm 2.5^\circ\text{C}$. Centrifuge the bottles at 3600 rpm for 25 minutes.

12.4.3 Empty the centrifuge bottles back into the recovery flask and add six 3-mm diameter glass boiling beads. Re-attach the flask to the rotary evaporator. Disconnect the transfer tube from the rotary evaporator and replace it with the gas purge tube. Disconnect the filtrate transfer line from the external rotary evaporator neck fitting and replace it with the nitrogen gas line. Apply 93.3 ± 0.7 kPa (700 ± 5 mm Hg) vacuum. Lower the flask approximately 38 mm into the oil bath.

12.4.4 Distill the solvent.

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