

GDT 119

A. Scope

For a complete list of GDTs, see the Table of Contents.

This method extracts asphalt cement from pavement or plant mix, then distills off the solvent to leave asphalt cement as residue. Test the residue using standard test methods for the various properties of the asphalt cement. This process enables determination of changes in the asphalt as it passes through an asphalt plant or as it remains on the roadway for various durations of time.

This method may involve hazardous materials, operations, and equipment and does not purport to address all of the safety problems associated with its use. Whomever uses this method must consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations before use.

Referenced Documents

1. AASHTO-T-164—Test Method for Quantitative Extraction of Bitumen from Paving Mixtures
2. Federal Specifications O-T-634—Reagent or Technical Grade Trichloroethylene
3. ASTM: D-2939—Method of Testing Emulsified Bitumens Used as Protective Coatings
4. AASHTO: T-179—Effect of Heat and Air on Asphalt Materials (Thin-Film Oven Test)
5. AASHTO T 315—Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).
6. AASHTO TP 70/ASTM D7405-Multiple Stress Creep Recovery Test

B. Apparatus

1. Rotavapor—Equipped with a distillation flask, a variable speed motor capable of rotating the distillation flask at a rate of 50 to 100 rpm, condenser, solvent recovery flask, and heated oil bath. Set the angle of the distillation flask from horizontal to the bath at approximately 59°. The distillation flask should have a capacity of 0.5 gal (2000 ml) and must be capable of being lowered to a depth of 1.5 in (38.1 mm). The Buch Rotavapor Model RE- 1 1 1 a and RE- 121 A are satisfactory.
 - A. Manometer or Vacuum Gauge—Device suitable for measuring and controlling the specified vacuum.
 - B. Gas Flowmeter—Apparatus capable of indicating a gas flow of up to 0.25 gal (1000 ml) per min.
 - C. Sample Container—Glass or plastic container having adequate volume to hold the sample and added solvent.
 - D. Vacuum System—Capable of maintaining a vacuum to within + 0.08 in (2 mm) of the desired level up to and including 20 in (500 mm) Hg.
 - E. Carbon Dioxide Gas—A pressurized tank with a pressure-reducing valve capable of delivering gas at a rate of 2 pts (900 ml) per minute.

- F. Solvent—Use reagent grade (preferred) or technical grade Trichloroethylene to extract asphalt from the aggregate and asphalt mixture. Use the procedure in AASHTO: T-164 Method E-1, conforming to the apparatus as shown in the procedure.
- G. Proof Testing—Determine the influence of the solvent on the recovered asphalt on each drum of solvent and periodically as appropriate to monitor the performance of the solvent. This is accomplished by mixing 0.25 lbs (100 g) of pretested asphalt cement into
- H. Prepare 0.5 gal (2000 ml) residue asphalt to define changes in its properties. When preparing the sample, heat the asphalt cement no hotter than 230 °F (110 °C) for no longer than 15 minutes. Thin film oven residue, or previously recovered asphalts will be satisfactory for this purpose.
- I. Sample Size and Preparation
 - Select The Sample of Mix
 - 1. Select a sample of the pavement or plant mix of such size to contain 1.8 to 2.6 oz (50 to 75 g) of asphalt cement.
 - 2. A mixture having $\geq 5\% < 6\%$ asphalt cement requires 2.6 to 3.3 lbs (1200 to 1500 g) of asphalt while a mix having $\geq 6\%$ should have 2.2 to 2.6 lbs (1000 to 1200 g).
 - 3. Where a mix has less than 5% asphalt, you may determine the sample size by dividing the anticipated percent asphalt into 7,500.

2. Vacuum Extraction Operation

- A. Select The Sample of Mix
 - 1. Select a sample of the pavement or plant mix of such size to contain 3.2 to 3.8 oz (100 to 120 g) of asphalt cement.
 - 2. A mixture having 5% asphalt cement requires 4.4 to 4.8 lbs (2000 to 2200 g) of asphalt while a mix having 6% should have 3.7 to 4.4 lbs (1670 to 2000 g).
 - 3. Where a mix has less than 5% asphalt, you may determine the sample size by dividing the anticipated percent asphalt into 10,000.
- B. Dry The Sample and Dissolve the Asphalt
 - 1) Crumble all aggregations in the sample and dry to a constant weight at a temperature not exceeding 230 °F (110 °C) for more than 1 hour.
 - 2) Place the sample into a gallon can, add approximately a quart of solvent, then place the lid securely onto the can.
 - 3) Shake the can from end to end or roll the can for 15 seconds every 5 minutes, or place the can without the lid into an ultrasonic bath and stir every 5 minutes.
 - 4) After 30 minutes pour off the solution and inspect the aggregate. Repeat this procedure until the aggregate is clean. You may use any alternate procedure that yields clean aggregate using minimal solvent without heat.
- C. Use the Vacuum Extractor

- 1) Gently pour the solution acquired into a vacuum extractor according to method E-1 of AASHTO: T-164.
- 2) Ensure that the apparatus used complies with AASHTO: T-164 and uses 8 oz (250 g) of diatomaceous earth. The filter support shall be No. 10 (2.00 mm) sieve made of brass or stainless steel to improve efficiency of the filtering operation.

D. Remove Remaining Mineral Matter

- 1) Remove mineral matter from the solution. The SMM (Spindle Motor Machine) Continuous Flow Filterless Centrifuge is satisfactory for this operation, however, exercise precautions to prevent water condensate from entering the solution during humid weather.
- 2) Determine ash content in the asphalt cement residue according to ASTM: D-2939, Method of Testing Emulsified Bitumen Used as Protective Coating.

C. Sample Handling and Precautions

Take the following precautions when using a solvent.

1. Toxicity—All operations involving the solvent should be used under a hood or with an effective surface exhaust system in a well ventilated area.
2. Corrosion—Trichloroethylene in the presence of heat and moisture may form acids that are extremely corrosive to certain metals, particularly when subject to frequent contact or over lengthy periods of time.
3. Decomposition—Trichloroethylene, when stored in a steel container and in continuous contact with moisture may decompose by dehydrohalogenation to form unsaturated hydrocarbon liquids and hydrogen chloride. Store steel drums containing trichloroethylene in a cool, dry location, and keep tightly sealed and opened as infrequently as possible. The hydrogen chloride in decomposed trichloroethylene may harden an asphalt during the extraction and recovery test. Once a steel drum is open, transfer the contents into brown glass jugs until it is used.
4. Oil Bath Protection—When the oil bath is not in use, cover it to prevent inadvertent entry of water. Water falls to the bottom of the bath and erupts violently at a temperature of about 350 °F (177 °C).

D. Procedures

1. Distillation Operation

A. Start Up

- 1) Heat the oil bath to 280 ± 5 °F (137.8 ± 3 °C) and lower the flask to touch the oil.
- 2) Start the flask rotation at 50 to 70 rpm.
- 3) Place the siphon tube into the demineralized solution and connect the other end to the pet cock. Align the pet cock hole to allow the vacuum to transfer the solution into the distillation flask above the oil bath.

B. Transfer Solution

- 1) Apply the 480 ± 2 °F (249 ± 1 °C) mm vacuum to siphon the solution into the distillation flask.

- 2) Start the cool water (55 °F (13° C) or colder) through the condenser coils.
 - 3) Maintain about 0.15 gal (600 ml) of solution in the distillation flask until the supply container is empty and the flask contains about 0.13 gal (500 ml) of solution. This should require about 15 minutes per quart of solution.
- C. Apply Carbon Dioxide
- 1) Remove the siphon tube.
 - 2) Attach the gas flow tube and start the flow of carbon dioxide at about 0.24 gal (900 ml) per minute. Continue this rate throughout the remainder of the distillation.
- D. Inspect Condenser
- 1) The solvent droplets will soon fall more slowly from the condenser tubes. As soon as they begin to slow down, increase the bath temperature to 360 °F (182 °C).
 - 2) Lower the flask into the oil bath to a depth of 1.5 in (38.1) mm, and increase rotation rate to 70 to 90 rpm.
 - 3) Do not allow the drip rate to slow prematurely to hinder removal of the solvent. Premature reduction of the drip rate may result in a more dense surface of higher viscosity that reduces solvent mobility.
- E. Inspect Asphalt Appearance
- 1) Ensure that the surface of the asphalt in the flask is smooth and free of bubbles, foam, or other surface imperfections.
 - 2) You may increase the flask rotation speed followed by slowing and another increase to dislodge any remaining solvent. Continue until the desired surface appearance is present but not longer than 25 minutes after the last drop is observed falling from the condenser tube.
- F. Shut Down
- 1) Turn off the vacuum, cooling water, and carbon dioxide and allow the flask to remain undisturbed for 2 minutes to allow the asphalt to collect.
 - 2) Raise the flask above the oil bath for 1 to 2 minutes to allow oil to drip from the flask but not cool significantly. Excessive cooling reduces the amount of asphalt that is poured from the flask.
- G. Transfer the Recovered Asphalt
- 1) Remove the flask and dry the oil from the flask with paper towels. Exercise care to avoid contaminating the asphalt with the oil.
 - 2) Without rotating the flask, pour the recovered asphalt into a 6 oz (170 g) cup or other desired container.
 - 3) You may place the flask into an oven at 325 °F (163 °C) for 15 minutes to cause the asphalt to flow more efficiently if needed. Ensure that this operation does not distribute the asphalt over the interior of the flask.
2. Testing the Recovered Asphalt Residue
- A. Test the Asphalt

- 1) Take portions of the recovered asphalt, while still in a liquid state, for penetration, softening point, ductility, ash content, kinematic and/or absolute viscosity, and other tests as desired. DSR testing in accordance with AASHTO T 315 may be performed in lieu of softening point, ductility, ash content, kinematic and/or absolute viscosity. When required by specifications, perform MSCR testing in accordance with AASHTO Tp70/ASTM D7405 with the following exception. When performing MSCR testing on recovered GTR modified asphalt cement obtained from mixtures produced from an asphalt plant, RTFO aging is not required.
- 2) If allowed to cool, place the sample in a 6 oz (170 g) penetration cup with the lid in place. Heat the cup in an oven at 325 °F (163 °C) for approximately 30 minutes to prepare for pouring the test specimens.
- 3) Conduct ash content determinations on recovered bitumens according to ASTM Method D 2939 and report with other test data for the recovered asphalt. Ash contents of recovered asphalt greater than 2% may affect the accuracy of the penetration, or viscosity tests.

E. Report

Report the viscosity, penetration, $G^*/\sin(\delta)$, phase angle, J_{nr} @ 3.2kPa, Percent Recovery @ 3.2 kPa and/or other test results according to the appropriate test method.