



Florida Method of Test for Recovery of Asphalt from Solution using the Rotavapor Apparatus

Designation: FM 3-D5404

1. SCOPE

- 1.1 This test method is intended to recover asphalt from a solvent using the rotavapor apparatus to ensure that changes in the asphalt properties during the recovery process are minimized.
- 1.2 This standard does not purport to address all the safety problems, 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.

2. REFERENCED DOCUMENTS

FM 5-524 Reflux Extraction of Bitumen from Bituminous Paving Mixtures.

3. SUMMARY OF TEST METHOD

- 3.1 The solution of solvent and asphalt from a prior extraction is distilled by partially immersing the rotating distillation flask of the rotavapor apparatus in a heated oil bath while the solution is subjected to a partial vacuum and a flow of nitrogen gas or carbon dioxide gas. The recovered asphalt can then be subjected to further testing as required.

4. SIGNIFICANCE AND USE

- 4.1 In order to determine the characteristics of the asphalt in an asphalt paving mixture, it is necessary to extract the asphalt from the aggregate by means of a suitable solvent and then to recover the asphalt from the solvent without significantly changing the asphalt's properties. The asphalt recovered from the solvent by this test method can be tested using the same methods as for the original asphalt cement, and comparisons between the properties of the original and recovered asphalt can be made.



5. APPARATUS

- 5.1 Rotavapor, equipped with a distillation flask, a variable speed motor capable of rotating the distillation flask at a rate of at least 80 rpm, condenser, solvent recovery flask, and heated oil bath. The angle of the distillation flask from the horizontal to the bath is set at approximately 15°. The distillation flask (see **Note 1**), when fully immersed, should be at a depth of approximately 1.5 in (38.1 mm).¹

Note 1: A flask having a 1000 mL capacity is recommended.

- 5.2 Manometer or Vacuum Gauge, suitable for measuring the specified vacuum.
- 5.3 Gas Flowmeter, capable of indicating a gas flow of up to 1000 mL/min.
- 5.4 Sample Container, having adequate volume to hold the sample and added solvent.
- 5.5 Vacuum System, capable of maintaining a vacuum to within 10 mbar of the desired level up to and including 210 mbar pressure.

6. REAGENTS AND MATERIALS

- 6.1 Nitrogen Gas or Carbon Dioxide Gas, pressurized tank with pressure-reducing valve, or other convenient source (see **Note 2**).

Note 2: Different flow rates may be required depending on whether nitrogen gas or carbon dioxide gas is used.

- 6.2 Oil: The oil for the heated oil bath should be Fisher Scientific with a flash point above 360°F (182°C) or an equivalent. The flash point is determined in accordance with ASTM D92.
- 6.3 Solvent: The solvent for extracting the asphalt form mixtures should be reagent grade trichloroethylene.²

¹ The Buchi Rotavapor RE-111A, or its equivalent, has been found satisfactory for this purpose. The Buchi Rotavapor is available from Fisher Scientific, Pittsburgh, PA.

² Trichloroethylene, Technical Grade, Type I (Federal Specification O-T-634, latest revision) may be used, but it is recommended that for each new supply of the solvent, a blank should be run on an asphalt of known properties.



7. PRECAUTIONS

- 7.1 **CAUTION:** The solvents listed in Section 6.3 should be used only under a hood or with an effective surface exhaust system in a well-ventilated area, since they are toxic to various degrees as indicated below.
- 7.2 These solvents in the presence of heat and moisture may be hydrolyzed to form acids that are extremely corrosive to certain metals, particularly when subject to contact over lengthy periods of time. Proper precautions should be taken to not allow these solvents to remain in small quantities in the effluent tanks of aluminum vacuum extractors.
- 7.3 Exposure of these solvents or their vapors to high temperatures such as contact with flames, hot glowing surfaces, or electric arcs can produce decomposition products such as hydrogen chloride. Steel drums containing these solvents should be stored in a cool, dry location, kept tightly sealed, and opened as infrequently as possible. The hydrogen chloride in decomposed solvent may harden an asphalt during the extraction and recovery test.

8. SAMPLE PREPARATION

- 8.1 The sample shall be obtained and handled in accordance with FM 5-524. This includes centrifuging the solution from a previous extraction for a minimum of 30 min at a minimum force of 770 times gravity in either wide-mouth bottles or centrifuge tubes.

Note 3: If a continuous centrifuge is used, the extract solution shall be charged at a rate not to exceed 150 mL/min, while the unit is operating at a speed calculated to produce a centrifugal force of not less than 3000 times gravity.

9. PROCEDURE

- 9.1 Heat the oil bath to a temperature of $285 \pm 5^\circ$ ($140.5 \pm 2.8^\circ\text{C}$). Circulate cold water through the condenser.
- 9.2 Apply a vacuum of $210 \text{ mbar} \pm 10.0 \text{ mbar}$ pressure and draw approximately 600 mL of asphalt solution from the sample container into the distillation flask by way of the sample line. Begin a nitrogen or carbon dioxide flow of approximately 500 mL/min through the system (see **Note 4**). Begin rotating the distillation flask (at approximately 60 rpm) and lower the flask into the oil bath. Initially the immersion depth of the flask will be determined by the

need to achieve a controlled solvent evaporation rate. The correct rate of evaporation can be observed as a steady controlled stream of condensed solvent being collected in the recovery flask.

Note 4: The vacuum and nitrogen or carbon dioxide flow values may need to be adjusted depending on the location. Also, low flow values are recommended at the beginning of the rotary process when the sample volume is large because of the possibility of back flow into the vacuum system. The possibility of back flow can also be minimized by regulating the depth of distillation of the flask.

- 9.3 When the amount of asphalt solution within the distillation flask appears low enough so that more solution may be added, discontinue the nitrogen or carbon dioxide flow. Draw the remaining asphalt solution from the sample container into the distillation flask and readjust the nitrogen or carbon dioxide flow (see **Note 5**).

Note 5: The equipment may be modified to allow a continuous flow of solution from the sample container into the distillation flask such that the volume in the distillation flask is maintained at approximately 600 mL. The nitrogen or carbon dioxide flow is not started until all the solution has entered the distillation flask.

- 9.4 When the bulk of the solvent has been distilled from the asphalt and no obvious condensation is occurring on the condenser, immerse the flask to the recommended maximum immersion depth of approximately 1.5 in. (38.1 mm). Increase nitrogen or carbon dioxide flow to approximately 600 mL/min and the spin rate of the distillation flask to about 80 rpm (see **Note 6**). A 1 to 2 min delay before applying the vacuum is recommended. Hold or reduce vacuum if foaming or a bubbly formation occurs. When foaming subsides apply a residual vacuum of 40 mbar (see **Note 7**). Maintain this condition for 20 ± 1 min.

Note 6: A faster flask spin rate exposes more surface area of the asphalt and, hence, dislodges more traces of solvent from the asphalt. The rotation speed of the flask may also be varied, or the flask rotation may be stopped to enhance the removal of solvent.

Note 7: Due to the cooling effect of the increased nitrogen or carbon dioxide flow, an increase in the temperature of the oil bath is generally needed to maintain a constant sample temperature. Experience has shown that a typical oil bath temperature of 315 ± 5 °F (157.2 ± 2.8 °C) is satisfactory for this purpose.



- 9.5 At the end of the 20 min. period, remove the distillation flask from the apparatus and wipe the flask clean of oil. Pour the asphalt into a proper size container. If needed, the flask may be inverted and placed into an oven at $325 \pm 2^{\circ}\text{F}$ ($163 \pm 1^{\circ}\text{C}$) for 10 to 15 min. to cause the asphalt to flow into the container. The asphalt should not be allowed to be distributed over the interior of the flask.

- 9.6 Portions of the recovered asphalt, while still in a liquid state, can be taken for penetration, softening point, ductility, and kinematic and absolute viscosity determinations as required.