FLORIDA DEPARTMENT OF TRANSPORTATION
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Florida Method of Test for Recovery of Asphalt from Solution using the Rotavapor Apparatus

Designation: FM 5-627

1. SCOPE

1.1 This test method is intended to recover asphalt binder from a solvent/asphalt binder solution using the rotavapor apparatus to ensure that changes in the asphalt binder properties during the recovery process are minimized.

1.2 <u>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.</u>

2. REFERENCED DOCUMENTS

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

3. SUMMARY OF TEST METHOD

The solution of solvent and asphalt binder 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 binder can then be subjected to further testing as required.

4. SIGNIFICANCE AND USE

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

5. APPARATUS

5.1 Rotavapor device (see **Note 1**), 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, having a capacity of 1000 mL when fully immersed, should be at a depth of approximately 1.5 in (38.1 mm).

Note 1: The Buchi Rotavapor R-300, or its equivalent, has been found satisfactory for this purpose.

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 40 mbar of residual absolute pressure.

5.6 Balance capable of measuring to an accuracy of 0.1 mg conforming with **AASHTO M 231** analytical balance class A.

5.7 Source of heat (electric hot plate) capable of maintaining a temperature of 400 ± 5°F (204 ± 2.8°C °C).

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 binder from mixtures should be reagent grade trichloroethylene (TCE). Type 1 technical grade trichloroethylene, (**Federal Specification O-T-634**) may be used, but it is recommended that for each new supply of the solvent, a blank should be run on an asphalt mixture 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

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.

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 ± 5°F (140.5 ± 2.8°C). Circulate cold water though the condenser.

9.2 Apply a vacuum to obtain a residual absolute pressure of 210 mbar ± 10.0 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 3**). 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 3: 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 4**).

Note 4: 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 80 rpm (see **Note 5**). 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 vacuum to obtain a residual absolute pressure of 40 mbar (see **Note 6**). Maintain this condition for 20 ± 1 min.

Note 5: 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 6: 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.

9.6 The recovered asphalt, while still in a liquid state, should have 2 to 3 g of liquid asphalt poured into container of known weight for performing a solvent check. The solvent check is required prior to further testing of the asphalt binder.

9.7 The steps involved in performing a solvent check are described below:

9.7.1 After pouring the solvent check sample, let the sample and container cool to ambient temperature. Record the weight of the sample in the container on an analytical balance. This weight is labeled as “Initial Weight”. The Initial Weight should be obtained within one hour after the extraction process is complete.

9.7.2 Place the uncovered container with the sample onto the hot plate at 400 ± 5°F (204 ± 2.8°C) for ten minutes to allow the TCE to sufficiently evaporate from the asphalt binder. After the ten-minute heating period, remove the container from the hot plate and allow it to cool to ambient temperature. Weigh the sample and container uncovered. This weight is labeled as “Final Weight”.

9.7.3 Calculate the Percent TCE Residue in the asphalt binder sample to the nearest 0.1%, as follows:

$$\text{Percent TCE Residue (\%)} = \frac{(\text{Final Weight} - \text{Initial Weight})}{\text{Initial Weight}} \times 100$$

9.7.4 If the Percentage TCE Residue is less than or equal to 0.4%, then testing of the remaining asphalt binder may be performed. If the Percentage TCE Residue exceeds 0.4%, then place the container with asphalt binder into an oven at 325 ± 5°F (163 ± 2.8°C) for 20 minutes. The container with asphalt binder should be uncovered in the oven to allow the TCE to sufficiently evaporate from the asphalt binder. Stir the asphalt binder after being in the oven for 10 minutes and then place the asphalt binder back into the oven for the remaining 10 minutes. Subsequently, pour another 2 to 3 g sample of asphalt binder into a clean container of known weight and repeat steps 9.7.1 - 9.7.4, up to a maximum of one additional reheat.

9.8 After confirming the Percentage TCE Residue is less than or equal to 0.4%, the recovered asphalt binder may be tested as needed.