FLORIDA DEPARTMENT OF TRANSPORTATION
State Materials Office
5007 NE 39th Avenue
Gainesville, Florida 32609
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Florida Method of Test
for
REFLUX EXTRACTION OF BITUMEN FROM BITUMINOUS PAVING MIXTURES

Designation: FM 5-524

1. SCOPE

This method covers a procedure for reflux extraction of bitumen in hot-mixed pavement mixtures and pavement samples to be used in the Recovery of Asphalt from Solution Using the Rotavapor Apparatus, FM 3-D5404.

2. REFERENCED DOCUMENTS

AASHTO Standard:

M 231 Weighing Devices Used in the Testing of Materials

Florida Method of Test:

FM 3-D5404: Standard Test Method for Recovery of Asphalt from Solution Using the Rotavapor Apparatus

3. SUMMARY OF METHOD:

The paving mixture is extracted with a suitable reagent grade trichloroethylene using the extraction equipment applicable to the particular method. The method described herein should not be used to determine gradation analysis or asphalt content of asphalt mixtures.

4. APPARATUS

4.1 Oven - Capable of maintaining a temperature at 110 ± 5°C (230 ± 9°F).

4.2 Balance - A balance conforming to the requirements of AASHTO M 231, Class G2. Balances with a greater degree of accuracy may be used.

4.3 Glass Jar - Cylindrical, Plain 8-3/4 ± 1/2 in. (222 ± 12.8 mm) OD, 18 " 2 in. (457 ± 25.6 mm) high, made of heat resistant glass. Open end of glass jar shall be ground smooth to insure a tight fit with the condenser.

4.4 Cylindrical Metal Frame - 7-1/2 ±1/4 in. (190.5 ± 6.4 mm) OD, and 9-1/4 ± 1/2 in. (235 ± 12.8 mm) high. The frame shall contain a wire formed or screened cone having a base of approximately 7-1/2 in. (190.5 mm) in diameter and side length

of approximately 7-1/2 to 8-1/2 in. (190.5 mm to 215.9 mm) mounted inside the top rim of each frame.

4.5 Condenser - 9 in. (229 mm) in diameter, the dimensions of such should insure a tight fit with the glass jar. The condenser shall have a truncated hemispherical or castellated condensing surface. It shall be equipped with approximately 1/4 in. (6.4 mm) ID tubing water inlet and outlet assembled so that no lead soldered joint comes in direct contact with the condensing solvent vapor.

4.6 Filter Paper - A fast filtering paper (500 mm in diameter) which will allow the solvent and bitumen to flow freely and not deteriorate the paper.

Note: A reinforced disposable Teri-Wiper manufactured by Kimberly-Clark Corporation, Stock No. 34865, has been found satisfactory for this purpose.

4.7 Insulating Pad - Approximately 1/8 in. (3.2 mm) thick.

4.8 Hot Plate - A 110 or 220 volt hot plate of sufficient size to accommodate the reflux jar and capable of gently boiling the solvent.

5. PROCEDURE

5.1 Place filter paper in cylindrical metal basket and open to form a hollow cone. If standard 500 mm filter paper is used, fold the filter on its diameter and fold twice again, one fold being made over the other to make three segments. Open to form a hollow three-ply cone with a single one-ply seam.

5.2 Select the test sample size to yield approximately 95 to 100 grams of recovered asphalt cement. This may be calculated based on the percent asphalt content in the sample to be recovered.

Sample size for 6.0% A.C.

Example: 95 grams - 6.0% A.C. x 100 = 1583 g

5.3 Deposit the sample in the filter paper-lined cone.

5.4 Pour approximately 500 ml to 600 ml as required by jar size, of reagent grade trichloroethylene into the glass jar and place the cone and frame with supporting legs in the jar. The solvent level must be below the tip of the cone.

5.5 Place the loaded jar on the electric hot plate and cover the jar with a condenser. Note: (Direct contact between the glass jar and the hot plate should be avoided by the use of an appropriate insulating pad.) Circulate a gentle steady flow of

cold water through the condenser. Adjust the heat so that the solvent runs into the sample in the cone. Take care to adjust the heat so that the filter cone does not overflow. Continue extraction until the solvent running from the tip of the cone appears a very light straw color when viewed against a white background. Shut off the heat but not the condenser water, and allow to stand until cool enough to handle.

5.6 Remove the frame with filter paper and extracted aggregate from the jar.

5.7 After the jar is cool enough to handle, transfer the extract to a graduated beaker.

5.8 The entire procedure, from the start of the reflux extraction to the final Rotavapor recovery (FM 3-D5404), must be completed within 8 hours.