FLORIDA DEPARTMENT OF TRANSPORTATION
State Materials Office
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Florida Method of Test
For
Extraction of Salts from Flexible Filler Material

Designation: FM 5-607

1. Scope

1.1. This method covers the extraction of chloride, sulfate, nitrate, and sulfide from wax material used to fill voided areas within post-tensioning ducts. The extract resulting from this method can be analyzed by other methods to determine the concentration of each constituent.

1.2. This method may be suitable for the evaluation of other flexible filler material. The user should evaluate safety concerns and chemical compatibility before attempting to use this method beyond the scope listed in Section 1.1.

1.3. This method was developed from test method NF M 07-23 (February 1969).

2. Equipment

2.1. Extraction Apparatus – see Figure 1

2.1.1. Reflux Condenser

2.1.2. Connecting hoses for water circulation

2.1.3. Joint Adapter with stopper

2.1.4. Round Bottom Boiling Flask, 500 ml

2.1.5. Heating Mantle, 500 ml

2.1.6. Various rings stands, clamps, and connectors

2.2. Boiling chips

2.3. Separator Funnel, 500 ml

2.4. Hot plate

2.5. Thermometer, readable to at least 100 °C

2.6. Beaker, 100 ml (2)

2.7. Beaker, 250 ml (2)

2.8. Graduated cylinder, 25 ml

2.9. Graduated cylinder, 50 ml

Figure 1 – Extraction Apparatus Example

3. Reagents and Standards

3.1. Toluene, nitration grade

3.2. Acetone, analytical grade

3.3. Ethanol, analytical grade

3.4. De-ionized water

4. Quality Control

4.1. Demonstration of Capability

4.1.1. Each analyst must perform a Demonstration of Capability (DOC) prior to reporting test data. The DOC must be documented. The Demonstration of Capability must be repeated if there is a change in personnel or method.

4.1.2. Select a sample of flexible filler or similar material with at least one extractable salt concentration above the detection limit of the analytical method of choice. Perform this extraction method, followed by an analytical method in duplicate. Calculate the relative standard deviation of results. Percent relative standard deviation (%RSD) must be ≤20%.

5. Procedure

5.1. Weight 40 ± 0.1 g of the sample into the boiling flask. Add the boiling chips to reduce bumping during the extraction. Heavier boiling chips, such as marble, have been found to work well.

5.2. In a fume hood, set up the extraction apparatus without connecting the boiling flask to the condenser.

5.3. Start the cooling water flow.

5.4. Slowly heat the flask until the sample melts.

Note 1: Consult the product data sheet for an estimate of the melting point temperature. The contents should not be heated beyond 65 °C. The safety of this method has not been evaluated for materials with melting points higher than 60 °C.

5.5. Maintain the temperature of the sample near the melting point so that it remains in the liquid state. In four separate beakers heat 110 ml of toluene to 60 ± 5 °C, 25 ml of ethanol to 40 ± 5 °C, 15 ml of acetone to 40 ± 5 °C, 125 ml of de-ionized water to 60 ± 5 °C.

5.6. Carefully add the pre-heated toluene to the boiling flask. Swirl continuously until the sample completely dissolves.

5.7. Without allowing the mixture to cool, carefully add the pre-heated ethanol and the pre-heated acetone to the boiling flask.

5.8. Attach the boiling flask to the condenser and bring the mixture to a rapid boil for two minutes.

5.9. Remove from heat. When boiling subsides, add 125 ml of warm de-ionized water.

5.10. Re-heat the mixture and bring to a boil again. Boil for one hour. For products with viscosities greater than 110 mm2/s at 40 °C, perform two successive one-hour extractions.

Note 2: The mixture is volatile and the range of proper operation is small. The apparatus must be carefully monitored throughout the boiling period.

5.11. Remove heat. When boiling subsides, transfer to a separator funnel.

5.12. Allow the mixture to separate into 2 phases. Draw off and filter the aqueous (bottom) layer through a slow filter paper.

5.13. The filtered aqueous material is now ready for testing by an appropriate analytical technique.