ALDOT-408
POLYMER CONTENT OF POLYMER MODIFIED PAVING GRADE ASPHALT BINDER USING INFRARED SPECTROPHOTOMETER

1. Scope

1.1 This test method is used to determine the percent of polymer concentration of a Styrene-Butadiene-Rubber, Styrene-Butadiene-Styrene or Styrene-Butadiene polymer in polymer modified asphalt binders.

2. Referenced Documents

2.1. AASHTO Standards

2.1.1. T 302, Polymer Content of Polymer-Modified Emulsions

2.2. Equipment Manufacturer Operations Manual

3. Apparatus

3.1 Fourier Transform Infrared Spectrophotometer

3.2 High speed shearing mixer

3.3 Heating coil with variable rheostat

3.4 Certified Thermometer 40°-580°F (4°-305°C)

3.5 Sodium chloride infrared window

3.6 10 ml pipette

3.7 Disposable eyedropper

3.8 50ml Erlenmeyer flask w/caps

3.9 Hi-temp vacuum oven

3.10 Calibrated Analytical balances, 0.02 g. accuracy

3.11 1,1,1 Trichloroethane, HPLC grade

3.12 Infrared heat lamp

3.13 Metal containers to mix binders and polymers

4. Preparation of Polymer Standards

4.1 *Liquid Polymer*:

4.1.1 Weigh 500.00 ± 0.01 grams binder neat into a container suitable for mixing

4.1.2 Heat the binder to 360 ± 5°F (182 ± 2°C). Maintain this temperature while adding polymer.

4.1.3 Mix seven standards containing from 1.0 percent to 4.0 percent polymer (rubber solids) in 0.5 percent increments.

4.1.4 Using either an eyedropper or pipette, add the calculated amount of polymer drop-wise to the binder with the mixer operating.

4.1.5 While the standards are still in a homogeneous state and liquid, remove 0.50 ±0.02 gram of polymer standard and place in a labeled vial with a leak resistant top.

4.1.6 For all seven standards, place the polymer-modified binder in a vacuum oven at 20-mm Hg vacuum at 220°F (104°C) for 2 hours to drive off any residual moisture.

4.1.7 After drying, remove the flask from the oven and cool to room temperature before making the dilutions necessary for infrared scanning.

4.2 *Solid Polymers*:

4.2.1 Weight 500.00 ± 0.02 grams binder into a container suitable for mixing.

4.2.2 Heat the binder to 360 ± 5°F (182 ± 2°C). Maintain this temperature while adding polymer.

4.2.3 Mix seven standards containing from 1.0 percent to 4.0 percent polymer (rubber solids) in 0.5 percent increments.

4.2.4 Add the calculated amount of polymer to the binder with the mixer operating.

4.2.5 While the standards are still in a homogeneous state and liquid, remove 0.50 ±0.02 gram of polymer standard and place in a labeled vial with a leak resistant top.

4.2.6 Place the polymer-modified binder in a vacuum oven at 20-mm Hg vacuum at 220°F (104°C) for 2 hours to drive off any residual moisture or weigh every 15 minutes for constant mass.

4.2.7 After drying, remove the vials from the oven and cool to room temperature before making the dilutions necessary for infrared scanning.

5. Preparation of Polymerized PGAB

5.1 Heat the binder to 360 ± 5°F (182 ± 2°C) and maintain this temperature while adding polymer.

5.2 Stir the sample while still hot to achieve homogenous state. After it is thoroughly mixed, remove and place 0.5 ± 0.02 gram in a 50 ml Erlenmeyer flask.

5.3 If there is water in the sample, dry it for 2 hours at 220°F (104°C) and 20 mm of Hg vacuum or until the water has been removed or weigh every 15 minutes for constant mass.

5.4 After drying, remove from the oven and cool the sample to room temperature before making the dilutions for infrared scanning.

6. Dilutions for Infrared Scan

6.1 Dilute sample one (1) part binder; twenty (20) parts 1,1,1 Trichloroethane.

6.1.1 Weigh 0.5g of polymer modified binder into a 50ml Erlenmeyer flask

6.1.2 Pipette 10 mls of 1,1,1 Trichloroethane into each flask

6.1.3 Shake the flask until the binder has dissolved.

7. Procedure

7.1 *Preparing Cell Plate:*

7.1.1 Use an eyedropper to place the dissolved mixture on a sodium chloride infrared window making sure the mixture completely covers the whole cell plate

7.1.2 Place the cell plate under an infrared heating lamp until the 1,1,1 Trichloroethane is dissolved off the cell plate (approximately 10 minutes).

7.2 *Analysis of the Sample*:

7.2.1 Place the cell plate into the holder and place the holder in the FTIR. Wait 10-15 minutes to allow the nitrogen purge system to evacuate the residual carbon dioxide and water in the holding compartment.

7.2.2 Scan from 4000 cm^{-1} to 400 cm^{-1}.

7.3 *Determining the Relative Peak Heights*:

7.3.1 Draw a new baseline under the peaks at 965 cm^{-1} for SBR and integrate the area (A1).

7.3.2 Draw a new baseline under the peaks at 699 cm^{-1} for SBS and integrate the area (A1).

7.3.3 Draw a new baseline under the peaks at 1370 cm^{-1} for binder peak and integrate the area (A2).

7.3.4 Ratio the polymer peaks (A1) versus the binder peak (A2).

NOTE: If more than one type polymer is used then the polymer peaks should be added together to express (A1).

7.4 *Unknown Determination*:

7.4.1 Plot the relative peak ratio (A1/A2) vs. the percent polymer. The first order curve that is generated is used to determine unknown percent polymer by ratio the relative peaks of the unknown sample.

8. Report

8.1 Report the values of polymer content to the nearest 0.1 percent.

8.2 All data will be forwarded to the ALDOT Bureau of Materials and Tests Asphalt Laboratory along with a statement of polymer content on company letterhead, signed by an authorized representative of the testing laboratory.