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DIVISION OF ENGINEERING SERVICES
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METHOD OF TEST FOR LATEX CONCENTRATION IN ASPHALT EMULSIONS

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read **SAFETY AND HEALTH** in Part 3 of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

SCOPE

This method determines the concentration of some polymer modifiers in asphalt emulsions. Two types of modifiers have been identified, Styrene-Butadiene Rubber (SBR) and Ethylene Vinyl Acetate (EVA). Using an infrared spectrophotometer, a thin film is cast and following Beer's Law, infrared absorption can be related to concentration of SBR or EVA. This test method is divided into the following parts:

1. Standards and Sample Preparation
2. Infrared Analysis
3. Safety and Health

PART 1. STANDARDS AND SAMPLE PREPARATION

Standards should be made with the same polymer and asphalt as the sample to be tested.

A. APPARATUS

1. Balance: capable of weighing to 200 grams in 0.01-g divisions.
2. Disposable Test Tubes: 20 × 150 mm.
3. #7 tapered cork stopper.
4. Teflon sheet.
5. Freezer: capable of maintaining 0°C.
6. Forced draft oven: maintained at 110 ± 5°C.
7. Metal paint cans, ½ pint cans have been found to be satisfactory.

B. PROCEDURE

1. Styrene-Butadiene Rubber

   a. For each standard weigh 100.0 grams of hot asphalt into a metal can.

   b. Add an appropriate amount of well-mixed SBR emulsion to yield blends of 1, 3, and 6 weight percent SBR in asphalt.

   c. Heat the mixture to 150°C and blend with a mechanical mixer until homogeneous and the water is driven off.

   d. Calculate the percentage of SBR in the asphalt as follows:

$$\frac{\text{(Weight SBR emulsion, grams) (A) (100)}}{\text{Weight asphalt, grams + (wt SBR emulsion)(A)}} = B$$

A = % SBR solids, ASTM Designation D-1417

B = % SBR in asphalt blend

e. Pour approximately 2 grams of hot material onto a Teflon sheet and place in freezer.

2. Ethylene Vinyl Acetate

   a. Heat asphalt and weigh 100.0 grams into metal cans.

   b. Add 1, 3, and 6 grams of EVA.

   c. Heat to 150°C on a hot plate and stir for 5 to 10 min until EVA is blended.

   d. Calculate the percentage of EVA as follows:

$$\frac{\text{(Weight EVA, g) } 100}{\text{weight asphalt, g}} = C$$

   C = % EVA in the asphalt blend

3. Sample

   a. Stir thoroughly and pour approximately 2 grams onto a Teflon sheet and place in oven until dry.

   b. Place Teflon sheet in freezer.

PART 2. INFRARED ANALYSIS

A. APPARATUS

1. Tetrahydrofuran (THF): reagent grade

2. Trichloroethylene: reagent grade

3. 10-mL pipette

4. Infrared spectrophotometer (IR). The Perkin-Elmer Fourier Transform Infrared Spectrophotometer, Model 1750 and Quant 3 software package have been found to be satisfactory.

5. Variable speed mixer.

6. Potassium chloride crystal (KC1), 1 inch in diameter by about ¼-inch thick.

B. PROCEDURE

1. Weigh 1.00 ± 01 g of frozen asphalt /polymer mixture into a test tube.

2. For SBR, pipette 10-mL THF into the test tube, stopper and shake until the asphalt mixture is dissolved. For EVA, use 10-mL trichloroethylene to dissolve the mixture.

3. Put 5 to 7 drops of the resulting solution onto a KC1 crystal and let dry. The same KC1 crystal should be used for the standards and test sample so differences between crystals will not interfere with quantification.

4. Place crystal into the IR, set the baseline at 80 % transmittance at 4,000 cm^{-1}, and record the spectra, save for analysis.

5. SBR and ethylene vinyl acetate are quantified as follows:

   a. SBR

      1. Draw a baseline from the maximum transmission point, to the left of the 967 cm^{-1}, peak at approximately 980 cm^{-1}, and parallel to the ordinate.

      2. Measure from this baseline to the points of maximum absorption at 967 cm^{-1}, (SBR) and 1,377 cm^{-1}, (asphalt).

      3. Divide the peak height of SBR(S) by asphalt (A), S/A.

      4. Using a least-squares calibration curve of S/A versus concentration prepared with three standards, 1 %, 3 %, and 6 % polymer by weight, determine the SBR concentration of the sample.

   b. Ethylene vinyl acetate

      1. Draw a baseline from the maximum transmission point to the right of the 1,738 cm^{-1} (EVA) peak at approximately 1,720 cm^{-1}, and parallel to the ordinate.

      2. Measure from the baseline to the points of maximum absorption at 1,738 cm^{-1} (EVA) and 1,605 cm^{-1}, (asphalt, A).

      3. Calculate the ratio EVA/A, and follow SBR procedure, Paragraph 4 above.

PART 3. SAFETY AND HEALTH

Users of this method do so at their own risk.

This method involves hazardous materials, operations and equipment. Exposure to tetrahydrofuran and trichloroethylene should be minimized and their use limited to an adequately ventilated hood. Extra precautions should be taken with tetrahydrofuran as it is extremely flammable, and with trichloroethylene, because of its cancer causing and reproductive toxicity. Trichloroethylene is listed in the California Administrative Code (CAC), Title 22, S12000, Chemicals Known to the State to Cause Cancer or Reproductive Toxicity (April 1, 1988). It is the responsibility of whoever chooses this method for use by their personnel to consult and establish appropriate safety and health practices prior to use. Those using this method do so at their own risk.

Testing personnel should be familiar with the Laboratory Safety Manual and the Series 400 Safety Precautions.

Use proper equipment to prevent contact with tetrahydrofuran and trichloroethane. Safety glasses, lab coat and correct gloves should be worn when using these chemicals. Dispose of waste solvents following State and Local regulations.

Be careful to prevent burns when heating the asphalt. Wear heat resistant gloves when handling the hot materials.

REFERENCES
ASTM Designations D-1417
Caltrans Laboratory Safety Manual

End of Text
(California Test 401 contains 3 pages)