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METHOD OF TEST FOR LATEX CONCENTRATION IN ASPHALT EMULSIONS

A. SCOPE

This method determines the concentration of polymer modifiers in asphalt emulsions. Two types of modifiers have been identified, Styrene-Butadiene Rubber (SBR) and Ethylene Vinyl Acetate (EVA). The infrared spectrum of polymer modified asphalt is obtained from a thin cast film of the material using an infrared spectrophotometer. The concentration of SBR or EVA in the polymer modified asphalt can be determined by relating the infrared absorption of specific spectral peaks to concentration - following Beer's Law. The procedure of this test method is divided into the following parts:

1. Standards and Sample Preparation
2. Infrared Analysis

B. REFERENCES

ASTM D-1417 Standard Test Methods for Rubber Latices - Synthetic
Caltrans Laboratory Safety Manual

C. PROCEDURES

PART 1. STANDARDS AND SAMPLE PREPARATION

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

1A. APPARATUS

1. Balance: capable of weighing to 200 g in 0.01 g divisions.
2. Disposable test tubes: 20 mm × 150 mm.
3. #7 tapered cork stopper.
4. Teflon sheet.
5. Freezer: capable of maintaining 0°C.
6. Forced draft oven: maintained at 110°C ± 5°C.
7. Hot plate and mechanical mixer.
8. Metal paint cans. (½ pint cans have been found to be satisfactory)

1B. PROCEDURE

1. Styrene-Butadiene Rubber

   a. For each standard, weigh 100.0 g 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 (solids) in asphalt.

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

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

$$B = \frac{\text{(Weight SBR emulsion, g) x (A)}}{\text{(Weight asphalt, g) + (Weight SBR emulsion, g) x (A/100)}}$$

   where:

   A = % SBR solids, ASTM D-1417

   B = % SBR in asphalt blend

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

2. Ethylene Vinyl Acetate

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

   b. Add appropriate amounts of solid EVA resin to yield blends of 1, 3 and 6 weight percent EVA in asphalt.

   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:

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

   where:

   C = % EVA in the asphalt blend

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

3. Sample (asphalt emulsion)

   a. Stir the sample thoroughly and pour approximately 4 g onto a Teflon sheet and place in 110°C ± 5°C oven to until dry.

   b. Place Teflon sheet in freezer.

PART 2. INFRARED ANALYSIS

2A. 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 test tube shaker/mixer.

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

2B. PROCEDURE

1. Weigh 1.00 g ± 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 KCl 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 the SBR procedure in Paragraph 4 above.

D. HEALTH AND SAFETY

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).

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.

It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Prior to handling, testing or disposing of any materials, testers must be knowledgeable about safe laboratory practices, hazards and exposure, chemical procurement and storage, and personal protective apparel and equipment.

Caltrans Laboratory Safety Manual is available at:

http://www.dot.ca.gov/hq/esc/ctms/pdf/lab_safety_manual.pdf

Users of this method do so at their own risk.

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(California Test 401 contains 4 pages)