

Standard Method of Test for

Polymer Content of Polymer-Modified
Emulsified Asphalt Residue and
Asphalt Binders

AASHTO Designation: T 302



1. SCOPE

- 1.1. This test method is used to determine the percent concentration of a Styrene-Butadiene-Rubber (SBR), Styrene-Butadiene (SB), or Styrene-Butadiene-Styrene (SBS) polymer in a polymer-modified asphalt binder or emulsified asphalt residue. By using the infrared spectrum and the principles of Beer’s Law, the polymer content of the asphalt material can be determined.
- 1.2. *This standard involves hazardous materials, operations, or equipment. This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. REFERENCED DOCUMENTS

- 2.1. *AASHTO Standards:*
- M 231, Weighing Devices Used in the Testing of Materials
 - T 59, Emulsified Asphalts

3. APPARATUS

- 3.1. *Balance*—Conforming to the requirements of M 231, Class G 1.
- 3.2. *Containers*—For blending the asphalt material with polymer—metal 1-pt (0.47-L) cans with lids are suitable.
- 3.3. *Variable-Speed Shearing Mixer.*
- 3.4. *Heating Coil with Variable Rheostat.*
- 3.5. *Thermometer*—For determining temperatures between 4 and 305°C (40 and 580°F).
- 3.6. *High-Temperature Vacuum Oven* (if necessary for removal of residual moisture).
- 3.7. *Infrared Spectrophotometer or Fourier Transform Infrared Spectrometer*—Capable of recording spectra in the 1800-to-600-cm⁻¹ region.
- 3.8. *Infrared Windows*—Polished sodium chloride or potassium chloride windows, or other suitable medium for casting of infrared specimens. If testing by use of the attenuated total reflectance (ATR) method, an ATR accessory and the appropriate crystal are necessary.

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3.9. *Testing by Solvent-Diluted Method:*

3.9.1. *Glass Vial with Leak-Resistant Top.*

3.9.2. *Eyedroppers.*

3.9.3. *Infrared Heat Lamp.*

3.9.4. *Solvent.*

Note 1—Trichloroethane, technical grade, and tetrahydrofuran (THF) have been shown to be suitable solvents that do not interfere with the infrared spectrum. Other appropriate solvents may be used.

3.10. *Testing by ATR Method:*

3.10.1. *Release Paper* (wax paper or other suitable medium).

3.10.2. *Spatula*—Appropriate for spreading hot asphalt binder or emulsified asphalt residue.

3.10.3. *Solvent*—Appropriate for removal of the test material from the infrared window.

4. PREPARATION OF CONTROL SAMPLES

4.1. Prepare a series of standards to cover the range from 0 to 5 percent weight-to-weight (w/w) polymer (rubber solids) according to Equation 1.

Note 2—The standards are prepared on a w/w percentage. This percentage is based on the solids weight of the polymer and the weight of the base asphalt binder or the emulsified asphalt residue. If the polymer is in an emulsified form or the standards are being made with an asphalt emulsion, an accurate determination of the rubber solids or percent asphalt residue must be made in order to prepare accurate standards.

4.1.1. This equation makes the assumption that there is no polymer contained in the base asphalt binder or emulsified asphalt (*A*) and that all solids in the polymer modifier are polymer (*D*):

$$E = \frac{(B)(A)(C)}{(100 - B)(D)} \quad (1)$$

where:

E = mass of polymer modifier, g;

B = percent polymer in the standard (e.g., 1, 2, 3, etc.);

A = mass of the base asphalt binder or emulsified asphalt, g;

C = percent asphalt residue (determined by T 59 for emulsified asphalt, otherwise 100); and

D = percent rubber solids in polymer modifier (determined for emulsified polymers, otherwise 100).

4.2. For each standard, add the calculated amount of asphalt binder or emulsified asphalt needed into a container suitable for mixing. Allow sufficient material to be able to thoroughly blend each standard.

4.3. Heat the asphalt binder until it is fluid at a temperature not to exceed 163°C (325°F). An emulsion should be heated to 82 ± 1°C (180 ± 2°F).

- 4.4. While maintaining heat and with the mixer in operation, slowly add the calculated amount of polymer modifier for each standard to the container of base asphalt binder or emulsified asphalt. Allow the materials to mix thoroughly.

Note 3—The addition of solid polymers may require extensive heating and shearing. A gradual addition of the material may be necessary. Addition of emulsified polymers in heated asphalt binders should be performed slowly to prevent boilover.

5. PREPARATION OF POLYMER SAMPLES AND TESTING BY SOLVENT-DILUTED METHOD

- 5.1. Heat the asphalt binder until it is fluid, at a temperature not to exceed 163°C (325°F). An emulsion should be heated to $82 \pm 1^\circ\text{C}$ ($180 \pm 2^\circ\text{F}$) while stirring it to achieve a homogeneous state.
- 5.2. After the sample is thoroughly mixed, place approximately 1 g of the sample in a glass vial.
- 5.3. If there is any residual moisture in the sample, place the vial in a vacuum oven set at approximately 20 mmHg and $104 \pm 2^\circ\text{C}$ ($220 \pm 4^\circ\text{F}$) until constant mass is achieved.
- 5.4. After drying, remove the vials from the oven and cool them to room temperature.
- 5.5. Dilute the contents of each glass vial with approximately 10 mL (0.3 oz) of the solvent.
- 5.6. Shake the vial until the sample is completely dissolved in the solvent.
- 5.7. Place drops of each solvent-diluted material on a prepared infrared window using an eyedropper. Ensure that an even layer is placed on the window.
- Note 4**—Five to nine drops of an asphalt material diluted 1 g to 10 mL (0.3 oz) on a 38-by-19-mm (1.5-by-0.75-in.) window has been shown to produce an adequate film thickness. Use of the ratio of the specified peak for the polymer to the peak for the asphalt negates any deviations due to film thickness.
- 5.8. Allow the solvent to evaporate at room temperature, or place the window under a heat lamp for approximately 10 min to drive off any residual solvent. Complete evaporation of any residual solvent will prevent the analysis of misleading spectra.

6. PROCEDURE—TESTING BY ATR METHOD

- 6.1. Heat the asphalt binder, or, heat the asphalt residue from evaporation (T 59) when testing emulsified asphalt, until it is fluid at a temperature not to exceed 163°C (325°F) while stirring it to achieve a homogeneous state.
- 6.2. Pour the heated material, spreading it with a spatula, on a piece of release paper cut slightly larger than the face of an ATR crystal. Use enough material to result in a layer that will cover the face of the crystal. A thin film will make cleanup of the crystal easier. An asphalt thickness on the paper of about 1 mm (0.04 in.) is sufficient.
- 6.3. Prepare an ATR crystal following the manufacturer's guidelines.
- 6.4. Allow the material to cool for several minutes prior to affixing the asphalt surface in direct contact with the top of the face of the prepared ATR crystal. Apply finger pressure to the top of the asphalt material to push out any air bubbles. The material must make complete contact with the crystal without any bubbles. Examination of the underside of the crystal may expose any bubbles or points of noncontact.

Note 5—Follow the manufacturer’s guidelines for treatment of the crystal. Placement of hot material or too much pressure on the face of the crystal may cause it to crack.

7. DATA COLLECTION AND ANALYSIS

7.1. Analysis of the Standards:

7.1.1. Using either the solvent-diluted or ATR method, acquire spectra on the prepared windows or crystal for each standard. Collection of a background spectrum is recommended prior to sample analysis.

7.1.2. Integrate the proper peak.

Note 6—The infrared spectrometer may be equipped with software that will quantify a specific peak or multiple peaks by using peak height or peak area. It may be necessary to correct the spectral baselines for offset or tilt.

7.1.2.1. For the solvent-diluted method, determine the absorbance at 965 cm^{-1} for the polymer modifier (SBR, SB, or SBS) and at 1375 cm^{-1} for the base asphalt binder or emulsified asphalt residue on each standard. Divide the value of the peak at 965 cm^{-1} (A_1) by the peak at 1375 cm^{-1} (A_2) to obtain the absorbance ratio of the relative peak values (A_1/A_2) of each standard. See the example in Figure 1.

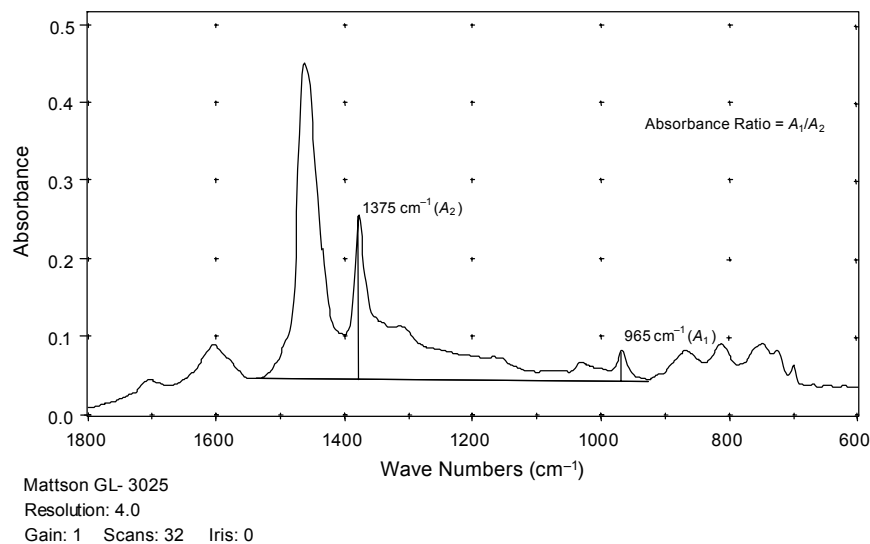


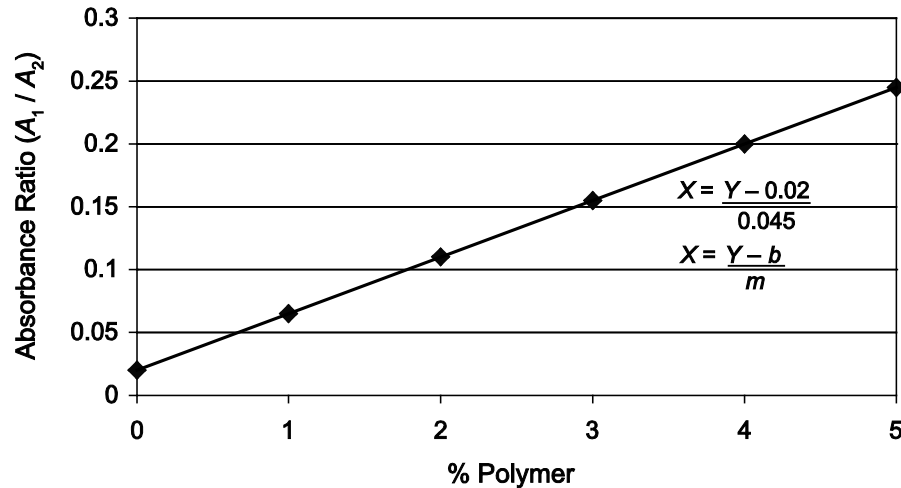
Figure 1—Infrared Scan of a Polymer-Modified Asphalt Material

7.1.2.2. For the ATR method, evaluate the peak area at 965 cm^{-1} for the polymer modifier (SBR, SB, or SBS). In the ATR method, the spectrum obtained is independent of the sample thickness, so it will not be necessary to integrate the peak at 1375 cm^{-1} .

7.1.3. Plot the absorbance ratio of the relative peak values (A_1/A_2) determined in the solvent-diluted method versus the corresponding polymer content or plot the peak area determined in the ATR method versus the corresponding polymer content, of each standard. Perform a first-order curve fit on the data to generate a standard curve. See the example for the solvent-diluted method in Table 1 and Figure 2.

Table 1—Typical Data for Standard Curve Development for Solvent-Diluted Method

Standard, % Polymer	Absorbance Ratio (A_1/A_2)
0	0.02
1	0.065
2	0.11
3	0.155
4	0.20
5	0.245

**Figure 2**—Polymer Content versus Absorbance Ratio (A_1/A_2)—Solvent-Diluted Method**7.2.** *Analysis of Sample:*

7.2.1. Analyze each sample using the applicable sections for the selected method (i.e., solvent-diluted or ATR) in Sections 7.1.1 through 7.1.2.2, and obtain the relative absorbance ratio (A_1/A_2) or peak area of the sample as appropriate.

7.2.2. Using the standard curve information, calculate the polymer content of the sample.

8. REPORT

8.1. The values determined for the polymer content shall be reported to the nearest 0.1 percent.

9. PRECISION AND BIAS

9.1. Information is currently not available to develop a precision and bias statement.

10. KEYWORDS

10.1. Emulsified asphalt residue; polymer content; polymer-modified asphalt binder; SB; SBR; SBS.