

Evaluation of Rheological Measurements for Unmodified and Modified Asphalt Cements

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A rigorous evaluation of the dynamic shear rheometer (DSR) with both unmodified and modified asphalts indicated that testing problems such as potential compliance-related problems, plate slip, and equipment limitations could be easily identified during testing. Once these problems were identified changes in either the test configuration or the testing parameters could eliminate the problem. The relationship between the complex modulus (G^*) and the complex viscosity (η^*) showed that G^* (in kilopascals) is equal to η^* (in poise) at 10 rad/sec. It was also shown that η^* , the DSR viscosity, could be estimated by $\eta(\dot{\gamma})$, the traditional vacuum viscosity for test temperatures of 46°C and above for unmodified asphalt cements. Typical values for the phase shift (δ) for test temperatures of 46°C and 64°C were 81 and 89 degrees, respectively, for unmodified asphalt cements and 60 and 81 degrees, respectively, for polymer-modified asphalts. For this range of test temperature the temperature had a greater influence on changes in δ than in the change of asphalt source or grade for unmodified asphalts. Changes in the phase shift for modified binders were dependent on the test temperature, type of polymer, and asphalt source or grade. A quick method of estimating the temperature at which $G^*/\sin \delta$ equals 1 kPa for unmodified asphalts is presented. Briefly, if $\sin \delta$ can be assumed to be 1 for unmodified asphalts, then the traditional viscosity measurements at both 60°C and 135°C can be used to estimate the temperature at which the viscosity would be 1000 P (i.e., $G^* = 1$ kPa). This approach compared well with the DSR-developed temperatures reported by Strategic Highway Research Program researchers.

Rheology is the study of material flow and deformation characteristics. Various methods of obtaining rheological measurements serve as the basis for the final Strategic Highway Research Program (SHRP) asphalt binder specification. A preliminary review of AASHTO test method TP5, Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (1), indicated that the percent strain, plate diameter, and gap height have been standardized for all binders. Since both the test method and the specification limits have been developed primarily from an evaluation of unmodified binders, a method of verifying the applicability of the specified test parameters should be added to the test method. This would ensure that all test results are as reliable as possible for a wide range of binders. Other critical information that has not been included in either TP5 or the supporting SHRP reports is a general frame of reference for reasonable ranges of values for complex modulus (G^*) and $\sin \delta$ (where δ is the phase shift), independent methods of quickly verifying the dynamic shear rheometer (DSR) results, and an estimate of the within-laboratory testing variability (2-4). A research program was developed to address these needs.

BACKGROUND

As stated in the introduction rheology is the study of both material flow and deformation. This is accomplished by measuring shear rate, shear stress, and in the case of oscillatory testing, the phase shift between the applied stress or strain and the corresponding response. Although most materials engineers in the highway industry equate a rheometer for measuring these properties with DSR, few people recognize that the traditional vacuum and kinematic viscometers are also rheometers. The vacuum viscometer is correctly classified as a pressure-driven, steady-state shear flow rheometer because the material moves through the capillary tube under a steady pressure. Information on the shear rate can be obtained from this test when Asphalt Institute tubes are used. The kinematic viscometer is also a pressure-driven, steady-state shear flow rheometer but can provide information at only a single, unknown shear rate. The rotational viscometer (e.g., Brookfield) used to determine the SHRP specification 135°C viscosity is classified as a steady-state drag flow rheometer.

The difference between the traditional and rotational rheometers and DSR (a dynamic drag flow rheometer) is that DSR also provides information on a material's storage and loss components. This information is especially important when binders are modified to increase their elastic properties.

It stands to reason that if the traditional vacuum viscometers and the new DSR are all rheometers, then the information obtained from each should be related. For low strain rates

$$\eta(\dot{\gamma}) = \eta'(\omega) \text{ for } \omega = \dot{\gamma} \rightarrow 0$$

where

$\dot{\gamma}$ = shear rate (sec^{-1}) (typical range for Asphalt Institute tubes is between 0.5 and 3 sec^{-1}),

ω = angular frequency (rad/sec),

$\eta(\dot{\gamma})$ = steady-state viscosity (e.g., vacuum viscosity measurement for unmodified asphalt cement), and

$\eta'(\omega)$ = dynamic loss component of viscosity.

For high frequencies an empirical relationship known as the Cox-Merz rule can be applied (5):

$$\eta(\dot{\gamma}) = \eta^*(\omega) \text{ for } \dot{\gamma} = \omega$$

Bouldin et al. (6) have shown that this relationship is generally applicable for both unmodified and polymer-modified asphalt cements.

Viscosity measurements are related to modulus by:

$$\eta^* = \frac{G^*}{\omega}$$

where η^* is the complex viscosity (in Pa · sec and, G^* is the complex modulus (in Pa). When ω is equal to 10 rad/sec, as specified in TP5, and viscosity is expressed in terms of poise (1 Pa · sec = 10 P), the equation reduces to

$$\eta^* = G^*$$

For Newtonian materials the viscosity will be independent of the shear rate. This means that for unmodified asphalt cements the traditional vacuum viscosity should be equal to G^* at 10 rad/sec.

For dynamic measurements both the complex viscosity and the complex modulus can be separated into loss and storage components by the following equations:

$$\text{Storage component: } \eta' = \frac{G'}{\omega}; G' = G^* \cos \delta$$

$$\text{Loss component: } \eta'' = \frac{G''}{\omega}; G'' = G^* \sin \delta$$

For materials with very low values of storage viscosity (η') the loss component of viscosity (η'') is essentially the complex viscosity (η^*). It should be noted that the superscripts for loss and storage for viscosity and modulus are traditionally reversed (i.e., η' and η'' and G'' and G' are loss and storage components, respectively).

RESEARCH PROGRAM

Objectives

The objectives of this research program were to

1. Develop a laboratory procedure to ensure the collection of reliable DSR data for both unmodified and modified asphalt cements.
2. Assess the applicability of the current version of TP5 for use with modified asphalt cements.

Scope

The hypothesis was that if the unaged, modified asphalt cements required changes in the test parameters, then the higher-viscosity aged materials would definitely need test method adjustments. The higher end of the temperature range in the SHRP specification is used to test either the original or rolling thin film oven (RTFO)-aged asphalt cements. All unaged materials were tested at these temperatures; a limited number of materials were selected for RTFO aging on the basis of the results for the unaged samples. Although the SHRP specification uses the lower test temperatures to evaluate pressure aging vessel-aged binders, this equipment was not available. Therefore, only unaged samples were tested at this temperature. When the TP5 test method was evaluated, single points obtained from frequency sweeps (0.1 to 100 rad/sec) were used.

Unmodified asphalt cements were chosen from the SHRP Materials Reference Library (MRL) and were selected to cover a wide range of viscosities. These binders included AC-10 (AAF-2), AC-20 (AAF-1), and 200/300 pen (AAA-2) asphalt cements.

Polymer modifiers were limited to the SBS (styrene-butadiene-styrene block copolymer) category. These included two different suppliers of SBS (Shell's Kraton, Dexco's Vector), two architectures (linear, radial), and four different molecular weights of saturated SBS [i.e., styrene-ethylene-butadiene-styrene, (SEBS)]. The concentration of polymer used for modifying the asphalt cement was limited to 4 percent; this was based on both a literature review and preliminary laboratory results.

TESTING PROGRAM

A Rheometrics RAA DSR was used to conduct frequency sweep testing at 5°C, 10°C, 15°C, 20°C, 25°C, and 30°C by using 8-mm-diameter plates and various gap heights ranging from 1.5 to 2 mm and at 20°C, 30°C, 40°C, 50°C, and 60°C by using 25-mm-diameter plates and gap heights ranging from 1 to 2 mm. The percent strains for each test temperature were based on the results of strain sweeps; strains in the middle of the linear viscoelastic region were selected. Additional single-point measurements were made for 46°C, 52°C, 58°C, and 64°C at 10 rad/sec and 12 percent strain. These single-point measurements were made in accordance with TP5.

Since G^* can be directly related to η^* and the dynamic and steady-state viscosities should be equivalent, traditional vacuum viscosities were also determined to confirm this relationship. This testing was completed at 60°C according to ASTM D2170.

All materials were tested in their unaged condition. For the polymer-modified asphalt cements this refers to samples molded immediately after the completion of blending as described in the next paragraph. After all unaged samples were tested, selected unmodified and modified binders were subjected to RTFO aging according to ASTM D2872. Materials were selected so that a wide range of both viscosities and polymer structures were represented. A discussion of the polymer structure is beyond the scope of this paper and will be mentioned only as a means of explaining specific differences in viscosity and modulus data.

For blending of the polymer-modified asphalt cements 4 percent polymer by weight of asphalt cement was added to approximately 450 g of preheated (between 175° and 185°C) base asphalt cement in a 1-L can. A high-shear blender set at 3,500 rpm was used to blend the polymer and the asphalt. A heating mantle was used around the can so that the asphalt temperature was maintained at the original temperature throughout mixing; the temperature was monitored throughout mixing. Blending was continued until Brookfield viscosity measurements (disk configuration) taken every 15 min either were consistent or began to decrease. Typical mixing times ranged from 45 min to 1.45 hr depending on the compatibility of the polymer with the asphalt cement.

Once blending was completed molds for the DSR were poured and the vacuum viscosities were determined.

ANALYSIS

Development of Guidelines for Quality Control of Data and Common Testing Problems

Quality can be corrupted by reporting data that have not been checked for evidence of potential compliance-related problems, plate slip, and equipment limitation problems. Potential compliance problems occur at cold temperatures; true compliance problems occur when the angular deflection produced by the motor is trans-

ferred into the deflection of the force transducer instead of the sample. Although true compliance may or may not be accounted for by the equipment software, other related problems such as exceeding a lower limit of angular deflection because of the stiffness of the material can be a part of this problem. Stiffness-related problems occur at different temperatures, plate sizes, and gaps for various materials. Plate slip occurs when the bond between the binder and the plate surface is lost and can also be a function of the sample preparation as well as the material stiffness. Since DSR takes only two measurements, torque and angular rotation, all results (e.g., strain, G^* and η^*) are calculated from these measurements. Therefore, it is essential that the limits for these measurements be carefully monitored.

Figure 1 shows how to identify stiffness-related problems. Figure 1 shows test results for AC-20 (AAF-1) modified with 4 percent of a radial SBS (Kraton D1184), the stiffest modified asphalt cement tested in the program described here. At 40°C the percent strain was very close to the target strain of 0.5 percent by using the 25-mm diameter plates. As the test temperature decreased it became increasingly apparent that the target strain could not be met. This is seen as a marked decrease in the measured percent strain and can be explained by the working equation used by the DSR:

$$\gamma = \frac{\theta R}{h}$$

where

- γ = strain,
- θ = angular rotation,
- R = radius of plate (mm), and
- h = gap between plates (mm).

The percent strain, gap height, and radius are entered into the equipment software by the operator at the beginning of the testing. However, there are limits on the minimum angular rotation that can be exceeded at a low strain level. Either the angular rotation

should be calculated to ensure that this is not a problem or the testing configuration should be changed to prevent the actual percent strain from differing from that for the target.

Plate slip or sample fracture occurring during a test can be identified by a sudden drop in the torque measurements. Plate slip due to improperly bonded samples can result in uniformly lower values. An indication of this problem can be seen when the individual curves forming the master curve do not overlap. When plate slip is suspected it can be easily identified by running a sample at either two different gaps or two different plate diameters (5,7). Figure 2 shows test results for unmodified AC-20 (AAF-1) from 25-mm-diameter plates with a gap height of 2 mm and 8-mm-diameter plates with a gap of 1.5 mm for a test temperature of 30°C. Both results overlapped very well between about 1 and 10 rad/sec.

Although plate slip is a potential problem that needs to be considered, no evidence of this problem was seen for any of the materials tested. This was attributed to the sample preparation procedure detailed in TP5 that specifies that the sample should be loaded onto preheated plates. It appeared that the preheating procedure effectively eliminates this problem.

Figure 2 also shows an apparent departure of results for the 25- and 8-mm-diameter plate results at the higher frequencies (above 10 rad/sec). An examination of the torque and percent strain values shows that this can be traced to stiffness-related problems (Figure 3). Because the percent strain decreased for the larger-diameter plates at the higher frequencies, the torque values did not increase as much as they should have. This resulted in the slightly higher viscosity for the 25-mm-diameter plates at the higher frequencies.

The erratic behavior for the 8-mm-diameter plate results at the lower frequencies can be traced to the fact that the limits of the measurement system were exceeded (Figure 3). For the Rheometrics RAA equipment the force transducer limits were a minimum of 2 g·cm and a maximum of 2000 g·cm. The manufacturer reported that although the minimum limit was 2 g·cm, reliable results may be obtained at as low as 0.2 g·cm. This appears to be the case in Fig-

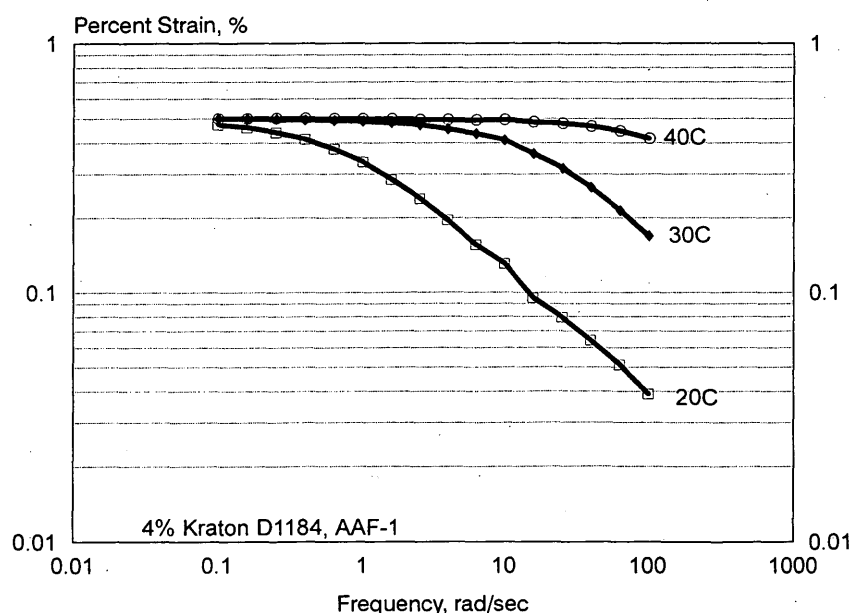


FIGURE 1 Identification of stiffness-related problems.

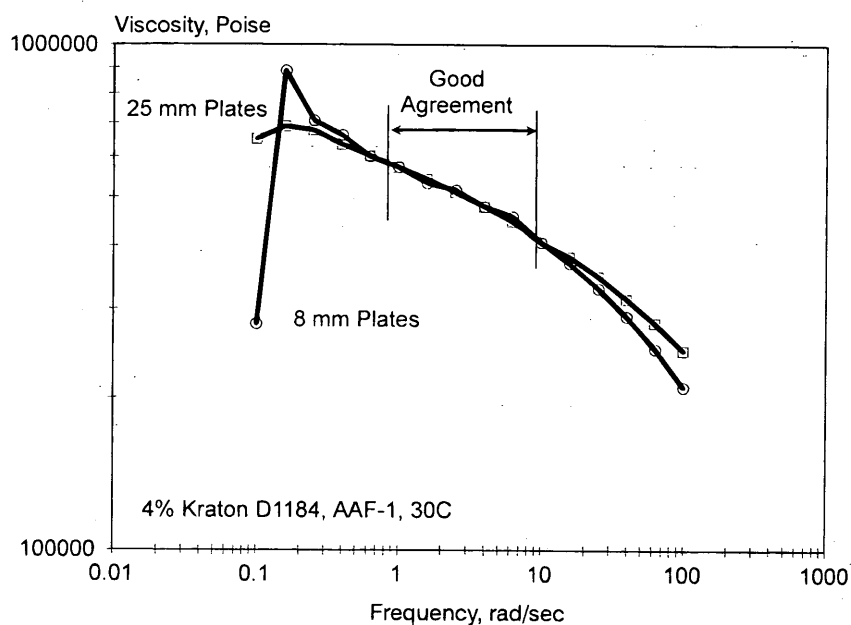


FIGURE 2 Identification of wall slip.

ure 3 because viscosity values above 0.2 g·cm provided a good overlap with the 25-mm-diameter plate data (Figure 2). However, torque values below 0.2 g·cm appear to be responsible for the erratic data seen in Figure 3.

Exceeding the maximum limit did not pose an identification problem with this equipment, because the equipment halts the testing when the maximum limit is reached.

Since DSR measurements rely solely on software to calculate test results, an additional manual check should be conducted to ensure that there are no software problems. This check has the additional

advantage of identifying another common problem: errors in geometry entered by the operator. Ordinarily, manual checking of software calculations can be too time-consuming and difficult for general laboratory personnel. However, since the basic working equations for DSR are simple, the manual check is also simple. Start by calculating the shear stress:

$$\tau = \frac{M}{2\pi R^3} \left(3 + \frac{d \ln M}{d \ln \dot{\gamma}_R} \right)$$

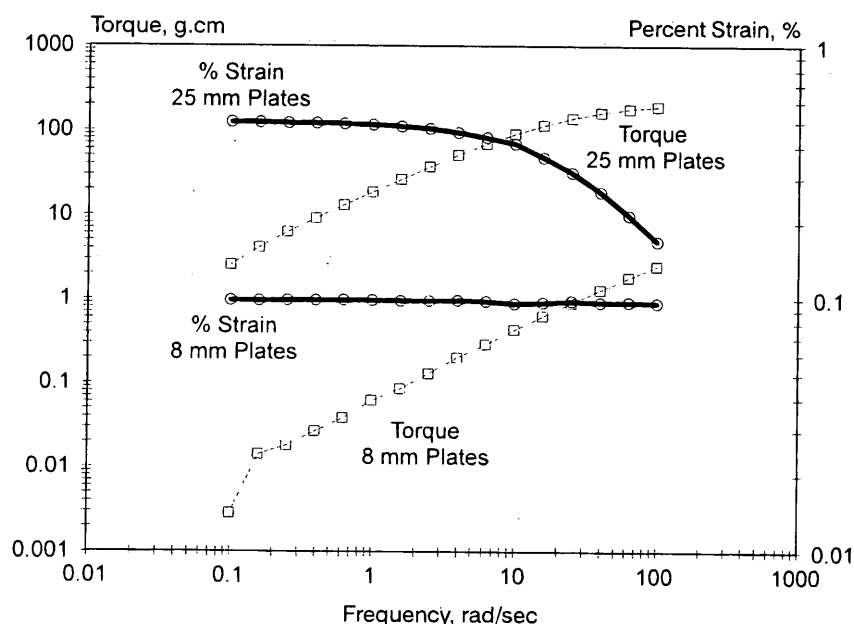


FIGURE 3 Identification of exceeded measurement limits.

TABLE 1 Solutions to Common DSR Problems

| Problem | Solution |
|-----------------------------|--|
| Stiffness-Related Problems | Reduce Plate Diameter Reduce Percent Strain |
| Plate Slip | Reduce Plate Diameter Reduce Percent Strain Check Sample Preparation |
| Max. Torque Limits Exceeded | Reduce Plate Diameter Reduce Percent Strain |
| Min. Torque Limits Exceeded | Increase Plate Diameter Increase Percent Strain |

where

M = torque value measured by equipment,

R = radius of plate (cm), and

$d \ln M / d \ln \dot{\gamma}_R = 1$ for measurements within the linear viscoelastic region.

Then calculate G^* :

$$G^* = \frac{\tau}{\gamma}$$

where γ is the software-reported strain. If the calculated value and the software-reported values do not match, there is most likely an error in the geometry values. The most common mistakes appear to be entering the plate diameter instead of the radius or forgetting to change the gap after loading a new sample.

Once compliance, plate slip, and limit problems have been identified they can easily be eliminated. Table 1 summarizes solutions

to these problems. As Table 1 shows all of the problems mentioned earlier can be addressed by changing either the plate diameter or the target percent strain. Compliance and related low angular deflections, slip, and upper-limit torque problems typically occur when the material is too stiff. The working equation for calculating the shear stress (presented earlier) explains why a change in the plate diameter will eliminate the problem. For a given level of shear stress the torque (M) decreases proportionally with the radius of the plates cubed (R^3). This also explains why increasing the plate size will eliminate any problems with low torque values.

Although these are very simple testing changes care must be taken to ensure that the values chosen are still within the linear viscoelastic region. This means that when one changes any of the testing parameters a strain sweep at a median frequency should be conducted. Figure 4 shows typical results from a strain sweep. Figure 4 shows that even the stiffest modified asphalt cement was still within the linear viscoelastic range for strains of up to 30 percent. These results indicate that there should be a wide latitude for changing the percent strain and still be within the linear viscoelastic region.

Evaluation of Test Parameters Specified by TP5

Once methods for identifying potential testing problems were developed, a range of both unmodified and modified asphalt cements was evaluated to check the appropriateness of the TP5 testing parameters. The specified testing parameters for original materials of 12 percent strain, 25-mm-diameter plates, 1-mm gap, and 10 rad/sec were appropriate for all materials tested. This conclusion was based on examining the torque and strain values at the warmest SHRP specification temperature (e.g., 46°C, 52°C, 58°C, or 64°C) at which a value of $G^*/\sin \delta$ was greater than or equal to 1 kPa. There were also no problems with testing RTFO-aged materials at a 10 percent strain level.

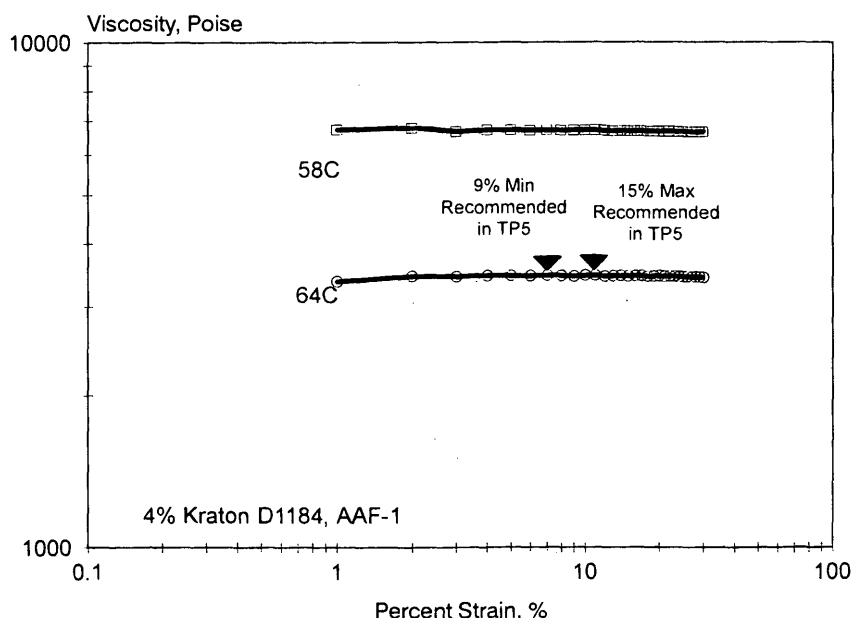


FIGURE 4 Strain sweep conducted to check for extent of linear viscoelastic region.

Changes had to be made for the unaged polymer-modified binders at the lower end of the 4°C to 40°C testing range. Typical strains used for the stiffer polymer-modified binders (AC-10 and AC-20 base asphalts) were between 0.05 and 0.2 percent for temperatures between 4°C and 19°C and between 0.5 and 1 percent for temperatures 22°C and 40°C.

Sample preparation also had to be changed for the modified asphalt cements at the colder temperature. The gap was set at the median of the test temperature range, the plates were heated to about 50°C, and the sample was loaded according to TP5. However, when the temperature was lowered back to the median temperature so that the sample could be trimmed, most of the polymer-modified asphalt cements showed a tensile stress buildup of greater than 1 kg or 50 percent of the capacity of the transducer. This was due to the quick drop in temperature (from 50°C to about 15°C in 3 min). The sample was contracting faster than it could dissipate the stress built up through a deformation response. To prevent the sample from debonding from the plates because of tension the operator manually decreased the gap height as the temperature dropped so that the force on the transducer was approximately zero. Once the sample and temperature stabilized, the edges of the sample were trimmed and the final gap distance was set.

Summary

The information presented on ensuring the quality of the reported data has been summarized into a flow chart for general laboratory

use (Figure 5). This flow chart for checking data is relevant either for single-point measurements used in the SHRP specification or in the development of master curves for research purposes.

One problem with evaluating test results was that there was no apparent means of assessing the reasonableness of the reported data. This led to the development of general ranges of material properties for common testing conditions. These ranges are given in Table 2. As expected the value of G^* reflects the range of viscosities of the various materials. The phase shift (δ), which is used to calculate $\sin \delta$, varied more with a 6°C change in test temperature than with a change in asphalt cement grade (unmodified) from what would be about an AC-5 to an AC-20. This difference decreased with increasing test temperature.

The RTFO aging of the unmodified asphalts significantly increased the G^* values; this reflects the increase in viscosity due to aging. The phase angle showed a slight, uniform decrease after aging for the lower test temperatures. This difference gradually diminished as the temperature increased.

There was little difference between either the source of the SBS product or the architecture of the polymer (i.e., linear versus radial). There was, however, an approximately 300 percent increase in the G^* values for a given asphalt cement source and grade due to the modification. The phase shift showed a significant increase with increasing test temperature and was also significantly influenced by the asphalt source or grade. In general, adding an SBS polymer resulted in a decrease in the phase angle from the mid to the high 80s to between 60 and the low 80s for unmodified materials. The

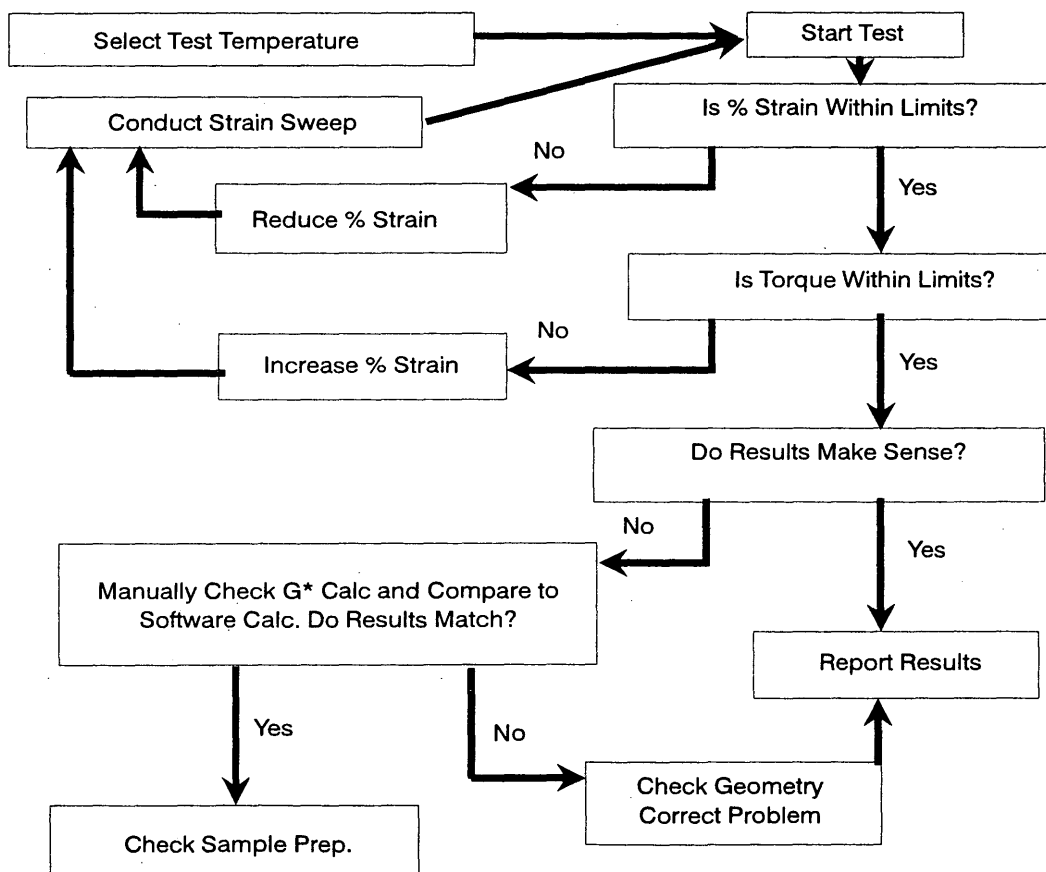


FIGURE 5 Quality control of reported data.

TABLE 2 Ranges of Rheological Parameters for Unmodified and Modified Asphalt Cements

| Category of Materials | Test Temp., °C | G*, kPa ¹ or η^* , Poise at 10 rad/sec. | | Phase Shift, δ^1 | |
|---|----------------|---|-----------------|---------------------------|--------------|
| Unaged, Unmodified Asphalt | 46 | 2,600 - (Not Available) | | 82.5 - 85.0 | |
| | 52 | 1,200 - 7,500 | | 85.4 - 86.6 | |
| | 58 | 594 - 2,755 | | 87.2 - 88.3 | |
| | 64 | 298 - 1,159 | | 88.5 - 88.9 | |
| RTFO Aged Unmodified | 46 | 27,180 ³ - 50,857 | | 81.1 - 82.5 | |
| | 52 | 9,003 ³ - 17,293 | | 84.1 - 85.5 | |
| | 58 | 3,303 ³ - 6,451 | | 86.3 - 87.2 | |
| | 64 | 1,328 ³ - 2,571 | | 87.7 - 88.5 | |
| Linear SBS Modified Asphalts 4% Polymer | 46 | Kraton D1101 | Vector 2518 | Kraton D1101 | Vector 2518 |
| | | 7,153 - 38,971 | 8,395 - 45,051 | 70.7 - 72.7 | 69.1 - 72.0 |
| | 52 | 3,605 - 15,764 | 4,106 - 18,251 | 73.46 - 75.2 | 71.2 - 73.2 |
| | 58 | 1,899 - 6,916 | 2,228 - 8,085 | 74.7 - 77.2 | 72.6 - 74.9 |
| | 64 | 1,026 - 3,211 | 1,155 - 3,844 | 75.0 - 77.7 | 73.5 - 75.7 |
| Radial SBS Modified Asphalts 4% Polymer | 46 | Kraton D1184 | Vector 2411 | Kraton D1184 ² | Vector 2411 |
| | | 7,722 - 39,033 | 9,451 - 33,156 | 68.6 - 72.7 (72.1) | 60.6 - 76.2 |
| | 52 | 4,002 - 15,693 | 5,182 - 12,843 | 73.7 - 75.17 (72.9) | 66.4 - 78.6 |
| | 58 | 2,071 - 6,887 | 2,826 - 5,375 | 76.9 - 77.2 (72.4) | 72.0 - 80.5 |
| | 64 | 1,073 - 3,344 | 1,490 - 2,415 | 78.8 - 77.7 (69.4) | 75.6 - 81.2 |
| SEBS Modified Asphalt 4% Polymer | 46 | Kraton G1652 | Kraton G1651 | Kraton G1652 | Kraton G1651 |
| | | 11,106 - 46,894 | 12,006 - 52,766 | 64.5 - 74.2 | 61.2 - 70.8 |
| | 52 | 5,708 - 18,893 | 6,508 - 21,632 | 68.6 - 75.9 | 65.6 - 72.2 |
| | 58 | 2,994 - 8,064 | 3,345 - 9,589 | 72.2 - 76.4 | 68.4 - 73.0 |
| | 64 | 1,553 - 3,826 | 1,675 - 4,594 | 76.0 - 73.0 | 72.2 - 73.0 |

- 1: The first value in each column is for the 200/300 Pen (AAA-2) asphalt and the second value is for the AC20 (AAF-1). The value for the AC10 (AAF-2) was always between these two values.
- 2: The values in parentheses are for the AC20 modified asphalt cement; the last number of this range is for the modified AC10 (AAF-2).
- 3: Data for AC10 (AAF-2) instead of AAA-2.

magnitude of the change was dependent on the source and structure of the polymer.

The same trends were also seen for the SEBS products of different molecular weights. The higher-molecular-weight Kraton G1651 (SEBS), which is 3.6 times the molecular weight of the G1652 (as reported by the supplier), increased G^* slightly, and there was a corresponding decrease in the phase shift. There did not appear to be any significant difference between the SEBS products, regardless of differences in molecular weight. The range of phase shift values was also lower for these saturated SBS products; these values ranged from 64.5 to 76.4 degrees.

Theoretically, the traditional vacuum viscosity should provide an excellent estimate of the DSR viscosity and hence G^* . Figure 6 shows a wide range of vacuum-versus-DSR viscosity results

(Table 3). Very good correlations were obtained for the unmodified asphalt cements and for the lower-viscosity modified binders (i.e., the modified AAA-2 series). This can be explained by verifying the Newtonian behavior of these materials (Figure 7). There was very little change in the viscosity with increasing shear rate for any of the unmodified asphalts, either aged or unaged. This implies that the vacuum tube viscosities with typical shear rates for Asphalt Institute tubes of between 0.5 and 3 sec^{-1} should represent viscosities at 10 sec^{-1} , the steady-state counterpart of the 10 rad/sec used for dynamic shear testing. As the non-Newtonian behavior of the modified asphalts increases, the vacuum viscosities at shear rates of less than 5 sec^{-1} showed a higher estimate of the dynamic viscosity at 10 rad/sec. For this reason the use of vacuum viscosities as a measure of G^* should be limited to unmodified asphalts. Any modified

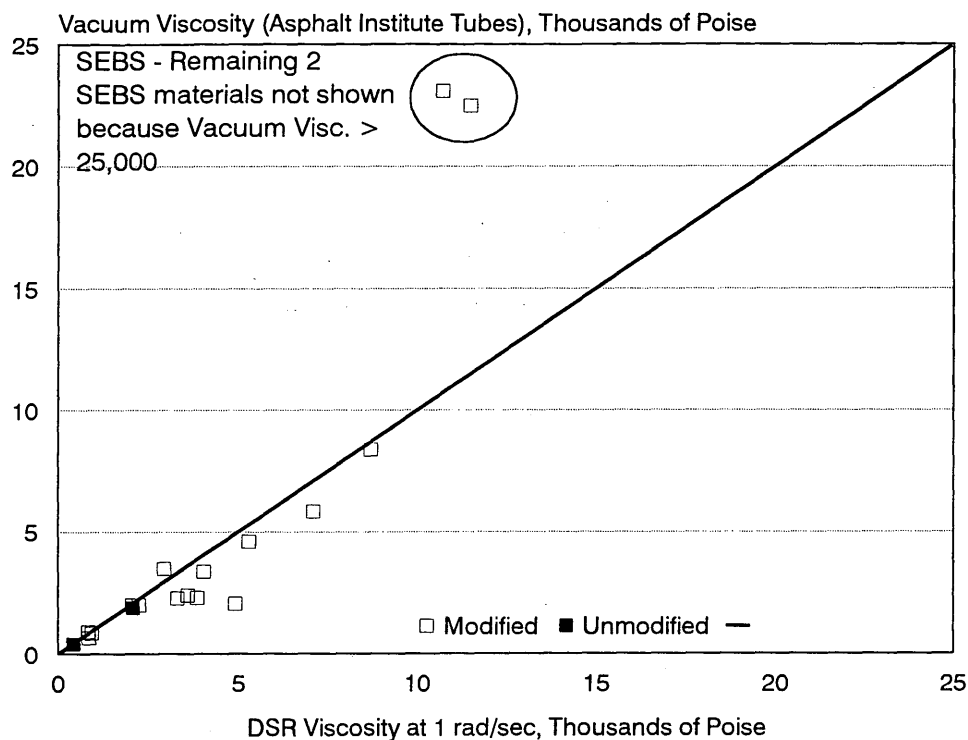


FIGURE 6 Comparison of vacuum and DSR viscosity versus shear rate results.

asphalts with undetermined shear rate-dependent behavior will be subject to an overestimation of G^* .

Table 2 shows that 4 percent Kraton D1184-modified AC-20 (AAF-1) behaved differently from the other modified asphalts. Although the viscosity decreased with increasing test temperature, the phase shift was essentially constant for all temperatures. It is possible that this phenomenon is a result of the highly developed polymer network formed within this asphalt. This network also appears to be responsible for the poor relationship between the vacuum and DSR viscosity (Table 3). This large difference between the viscosities can be explained in part by a closer examination of the vacuum viscosity data.

The DSR data exhibited shear thinning behavior typical of non-Newtonian materials. The vacuum tube results showed just the opposite behavior; the material appeared to be shear thickening. A closer examination of the theory behind capillary viscometers revealed that there is an assumption of fully developed shear flow within the measurement area (5). Before the full development of shear flow a region of extensional flow exists in all capillary rheometers. If the shear flow is not fully developed extensional thickening characteristics can show up as apparent shear thickening with highly networked materials. The increased viscosity with increased shear rate is due to the material stiffening response for the faster extensional flow. On the basis of this observation vacuum

TABLE 3 Comparison of Traditional Vacuum and DSR Viscosity Measurements

| Asphalt Cement | Modifier | Vacuum Viscosity Measurement ¹ (Poise) | DSR Viscosity Measurement (Poise) |
|---------------------|------------------------------|---|-----------------------------------|
| 200/300 Pen (AAA-2) | 0% | 410 | 439 |
| | 4% Kraton D1101 (Linear SBS) | 2,010 | 2,271 |
| | 4% Kraton D1184 (Radial SBS) | 2,160 | 2,078 |
| | 4% Vector 2411 (Radial SBS) | 3,495 | 2,976 |
| AC20 (AAF-1) | 0% | 1,872 | 2,089 |
| | 4% Kraton D1101 (Linear SBS) | 5,834 | 7,097 |
| | 4% Kraton D1184 (Radial SBS) | 201,630 | 9,344 |
| | 4% Vector 2518 (Linear SBS) | 8,723 | 8,370 |
| | 4% Vector 2411 (Radial SBS) | 4,935 | 2,056 |

1: Asphalt Institute tubes were used so that the shear rate for a given timing mark could be calculated. The vacuum tube viscosity and the DSR viscosity were compared at a shear rate of 1 sec^{-1} .

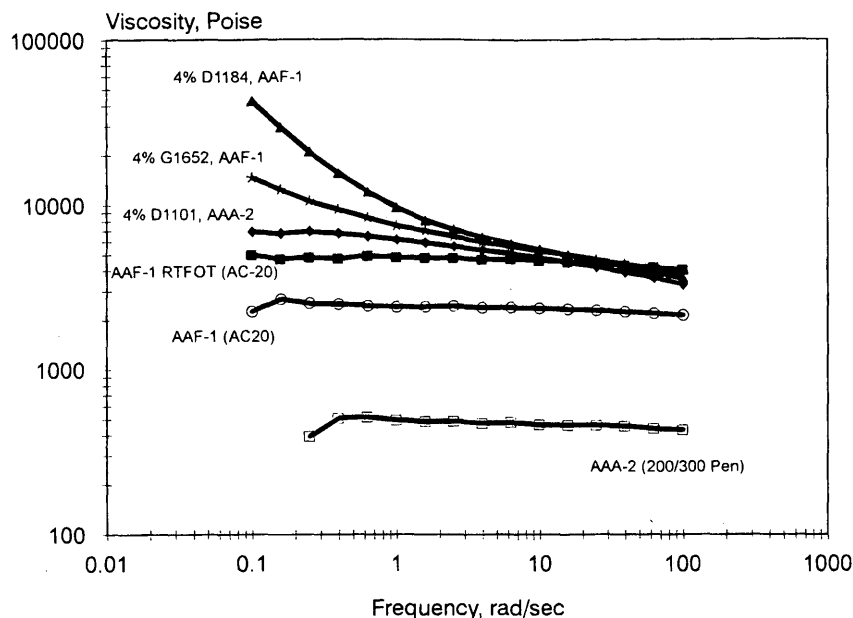


FIGURE 7 Viscosity versus shear rate for various materials.

viscosity results of modified asphalt cements should be used with caution and only as general estimates of the true material viscosity and G^* values.

Traditional viscosity measurements can be used to estimate not only G^* values for checking DSR results for unmodified asphalts but also the SHRP specification grade for an unclassified asphalt cement. Figure 8 compares the results of using the 60°C and 135°C viscosity data reported by the SHRP MRL to estimate the temperature at which $G^*/\sin \delta$ equals 1 kPa and the DSR-determined temperatures reported by SHRP researchers (2). $G^*/\sin \delta$ was estimated from the traditional viscosity data plotted on the Shell Bitumen Test Data Chart. The actual equations for the line for these plots were calculated to provide a more accurate estimate than just the graphical approach. Since δ only varied from about 81 to 89 degrees, which translated into $\sin \delta$ values from 0.987 to 0.999, an assumption of $\sin \delta$ equal to 1 was considered close enough for a reasonable estimate. Since the specific gravities of the asphalts were not reported in the SHRP MRL information, the kinematic viscosities were converted from centistokes to centipoise by assuming a constant specific gravity of 1.000.

Figure 8 shows that this quick approach to estimating the temperature at which a material has a $G^*/\sin \delta$ of 1 kPa is very good ($r^2 = 0.87$). This approach, however, tends to increasingly underestimate the temperature for increasing higher-viscosity asphalt cements. This is most likely due to the assumption of a constant 1.000 for specific gravities converting the kinematic results to poise. Generally, as the viscosity increases the specific gravity also increases; this would result in an increasing underestimation of the kinematic viscosity.

CONCLUSIONS

The following conclusions can be drawn from the results presented here:

1. Testing problems such as stiffness-related problems, plate slip, and exceeding the equipment limits can influence the quality of the reported test results. However, these problems are easily identified and corrected with changes in either the plate diameter or the specified percent strain.

2. The linear viscoelastic region of even the stiffest polymer-modified asphalt cement extended to at least 30 percent strain for temperatures as low as 58°C.

3. It appears that for test temperatures of between 4°C and about 30°C the AASHTO TP5 test parameters will have to be adjusted for polymer-modified asphalt cements. Some changes that are indicated include a reduction in the target percent strain and changes in sample preparation procedures.

4. G^* at 10 rad/sec is exactly equal to η^* . Since most asphalt cements at warmer temperatures (i.e., $> 48^\circ\text{C}$) are Newtonian, their viscosities are not shear rate dependent. Hence, typical values for G^* can be approximated by measuring the viscosity of the asphalt cement with conventional vacuum tube viscometers.

5. It is not recommended that vacuum viscosities be used to estimate G^* for modified asphalts because of their distinct non-Newtonian behavior.

6. Typical δ values for unmodified and RTFO-aged asphalts ranged from about 81 to 89 degrees for test temperatures between 46°C and 64°C. These values were more dependent on the test temperature than the grade of asphalt cement.

7. Typical phase shift values for modified asphalts ranged from 60 to 81 degrees for test temperatures of between 46°C and 64°C and were dependent on the type of polymer, the source or grade of the asphalt cement, as well as the test temperature.

8. A range of $\sin \delta$ from 0.987 to 0.999 appears to be reasonable for unmodified asphalt cement for test temperatures of between 46°C and 64°C. Therefore, an assumption was made that the viscosity (in poise) was equal to G^* (in kilopascals). Traditional viscosity data for 60°C and 135°C test temperatures were used along with the Shell viscosity-temperature graph to estimate the temperature at which the viscosity (i.e., $G^*/\sin \delta$) would be equal to 1 kPa (the SHRP mini-

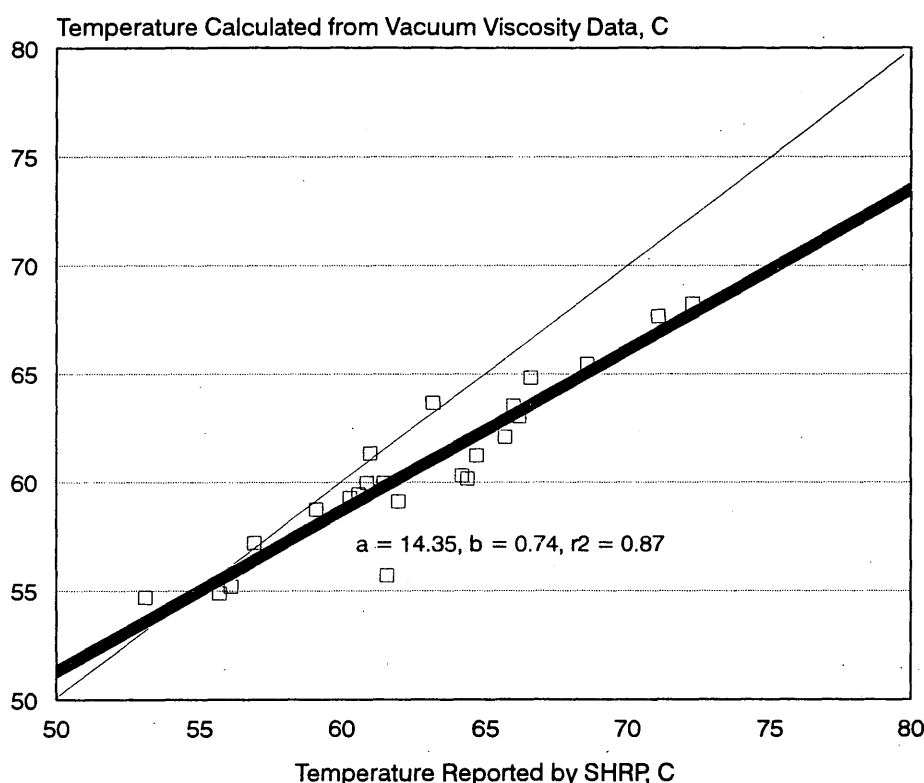


FIGURE 8 Estimating temperature at which $G^*/\sin \delta$ is equal to or greater than 1 kPa.

mum requirement for original binders). This method of estimating proved very good, with only a slight tendency to underestimate the SHRP-reported values reported for DSR results. The tendency to underestimate DSR results increased with increasing viscosity; however, the worst comparison was only 4°C too low.

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REFERENCES

1. Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer. AASHTO Test Method TP5. AASHTO, 1993.
2. Petersen, J. C., R. E. Robertson, J. F. Branthaer, P. M. Harnsberger, J. J. Duvall, S. S. Kim, D. A. Anderson, D. W. Christiansen, and H. U. Bahia. *Binder Characterization and Evaluation*, Vol. 1. SHRP Report SHRP-A-367. SHRP, National Research Council, Washington, D.C., 1994.
3. Anderson, D. A., D. W. Christiansen, H. U. Bahia, C. E. Antle, and J. Button. *Binder Characterization and Evaluation*, Vol. 3. *Physical Characterization*. SHRP Report SHRP-A-369. SHRP, National Research Council, Washington, D.C., 1994.
4. Petersen, J. C., R. E. Robertson, J. F. Branthaer, P. M. Harnsberger, J. J. Duvall, S. S. Kim, D. A. Anderson, D. W. Christiansen, H. U. Bahia, R. Dongre, C. E. Antle, M. G. Sharma, and J. Button. *Binder Characterization and Evaluation*, Vol. 4. *Test Methods*. SHRP Report SHRP-A-370. SHRP, National Research Council, Washington, D.C., 1994.
5. Macosko, C. W., R. G. Larson, T. P. Lodge, J. Mewis, and M. Tirrell. *Rheology: Principles, Measurements and Applications*. 1994.
6. Bouldin, M. G., J. H. Collins, and A. Berker. Rheological and Microstructure of Polymer-Asphalt Blends. *Rubber Chemistry and Technology*, Vol. 64, 1991.
7. Collins, J. H., M. G. Bouldin, R. Gelles, and A. Berker. Improved Performance of Paving Asphalts by Polymer Modifications. *Journal of the Association of Asphalt Paving Technologists*, Vol. 61, 1991.