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Improving Testing Requirements in Item 300 of TxDOT Standard Specification

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Improving Testing Requirements in Item 300 of TxDOT Standard Specification:

Final Report 0-7073

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Final Report

By

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16. Abstract Current test procedures in TxDOT specification Item 300, "Asphalts, Oils, and Emulsions" were reviewed for relevance, applicability, accuracy, and practicality, and recommendations were made for improving the tests and specifications. New test procedures were developed, as identified, to fulfill needed changes. Data for new proposed test procedures was generated and analyzed to develop suggested specification limits. This data explored the agreement between laboratories, developed test conditions, and facilitated round-robin experiments. Suggested changes were made for each table of the material specifications. A new long-term aging test was developed for PG binders.		
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Executive Summary

The goal of this project was to identify and update the tests and specifications in Item 300 as needed to ensure that the test methods and specifications are relevant, not redundant, and up-to-date.

To that end, the current test procedures were reviewed for relevance, applicability, accuracy, and practicality, and recommendations were made for improving the tests. Recommended changes along with the adoption level were developed.

New test procedures were developed, as identified, to fulfill needed changes.

Data for new proposed test procedures was generated and analyzed to develop suggested specification limits. This data explored the agreement between laboratories, developed test conditions, and facilitated round-robin experiments.

For the 2024 Item 300 (with inclusion of existing Special Provision 300- 003), suggested changes were made for each table of the material specifications.

Additionally, an extra study of binder aging of PG binders was performed. This includes short-term aging and PAV20, which are currently included in the specifications, and even longer-term aging termed PAV40, as well as a new procedure termed Thin Film PAV20. The Thin Film PAV20 was found to produce similar binder aging as the PAV40, but only required 20 hours of aging time. This Thin Film PAV20 is outlined in a proposed test procedure, based on the Pressure Air Vessel with modified test and specimen conditions.

This entire work is presented for evaluation and possible update of Item 300, “Asphalts, Oils, and Emulsions.”

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Chapter 1. Introduction

TxDOT standard specification Item 300, "Asphalts, Oils, and Emulsions", includes more than 48 different test procedures or conditioning procedures for Asphalt Cement (AC) graded binders (12), cutbacks (8), emulsified binders (17), crack sealers (3), asphalt rubber binders (3), recycling agent, and Performance Graded (PG) binders (11+) used in different pavement construction and maintenance applications. This does not include testing requirements for recycling agents, crack sealants, or asphalt rubber binder. TxDOT routinely performs many of these tests on a regular basis for quality management or quality assurance purposes. This presents a challenge for the binder lab to maintain test procedures, acquire and maintain test equipment, calibrate equipment, and train personnel. Some specification tests are legacy tests that once were the state-of-the-art and thought to be related to asphalt binder performance. However, many of these legacy tests are no longer used in more recently developed specifications as other tests are now available that are better correlated with performance.

1.1 Study Objectives

The main goal of this project is to update Item 300, as needed, to ensure that the test methods and specifications are relevant, not redundant, and up-to-date. In order to achieve this goal, each test in the Item 300 specifications was reviewed for relevance, applicability (i.e., whether the test was intended to assess safety, performance, or constructability), accuracy (i.e., ability to predict intended parameters), and practicality (i.e., cost of capital equipment; time taken for sample preparation and other activities before, during, and after the test; and environmental considerations such as use of solvents). The test data from a 12-month window from the TxDOT LIMS (Laboratory Information Management System) was also reviewed to identify current workload and consequently areas for prioritization.

1.1. Organization

This report includes seven chapters. Chapter 1 provides problems statement and objectives. Chapter 2 presents the background on the tests included in Item 300 specifications. It also categorizes the recommended changes in Item 300 based on the level of adoption. Chapter 3 compiles the new test methods developed during this project which includes step-by step procedures. Chapter 4 presents the data collected for the new test methods developed during this research. Chapter 5 presents the revised Item 300 specifications. Chapter 6 presents an extension of aging testing to investigate a long-term aging procedure. Chapter 7 is a short summary and conclusions of the overall research.

Chapter 2. Background

This chapter consists of a summary from the literature review of the different tests included in Item 300. A classification of these tests based on the general purpose of the tests or the type of materials they cover is presented in Figure 2.1 below. Tests were reviewed for relevance, applicability, accuracy, and practicality, and recommendations for improving the tests. A detailed review of each test is presented in this chapter. After taking the pros and cons of each test, as well as input from TxDOT and industry, into consideration, the changes to the current Item 300 specifications were proposed. These tests have been categorized on the basis of level of adoption and have been summarized towards the end of this chapter.

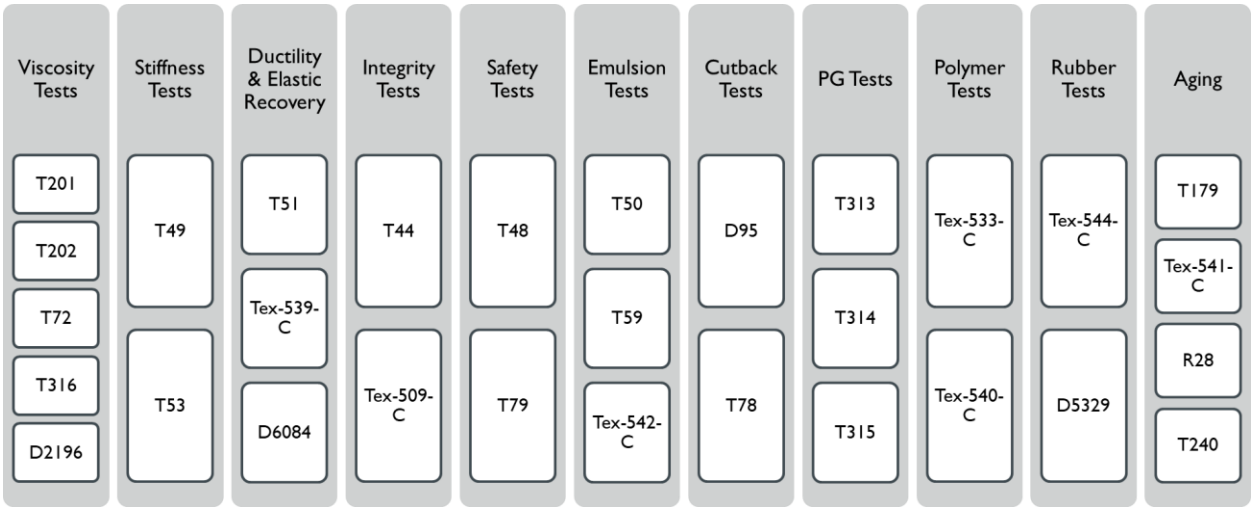


Figure 2.1. Classification of Item 300 Tests.

2.1. Viscosity Tests

2.1.1. AASHTO T201 "Standard Method of Test for Kinematic Viscosity of Asphalts (Bitumens)" and T202 "Standard Method of Test for Viscosity of Asphalts by Vacuum Capillary Viscometer"

2.1.1.1. Other Equivalent or Closely Similar Standards

T201

- ASTM D2170

T202

- ASTM D2171

2.1.1.2. Applicable Materials and Test Conditions

T201 uses:

- RC, MC, and Special-Use Cutbacks (Tables 4, 5, 6) at 140°F

T202 uses:

- AC, Polymer-Modified AC, (Tables 2, 3) at either 140°F (AC Only: TFOT residue; AC) OR 275°F (AC and Polymer-Modified AC both: Unaged)
- RC and MC Cutback, Polymer-Modified Emulsions and Cationic Emulsions, Specialty Emulsions (Tables 4, 5, 9, 10) at 140°F (distillation residue)

2.1.1.3. Utility and Background of Test

T201

This test method measures the kinematic viscosity of liquid asphalts, road oils, and distillation residues of liquid asphalts, all at 140°F, and of liquid asphalt binders at 275°F. It is applicable to asphalt binders having viscosities in the range from 30 to 6000 cSt.

In summary, this test measures the time required for a fixed volume of the liquid to flow through the capillary of a calibrated glass capillary viscometer under an accurately reproducible head and at a closely controlled temperature. The kinematic viscosity in cSt is then calculated by multiplying the efflux time in seconds by the viscometer calibration factor.

T202

This test method measures the apparent viscosity of asphalt binders at 60°C. It is applicable to asphalt binders having viscosities in the range 0.036 to over 200 000 P.

In summary, this test measures the time required for a fixed volume of a liquid to be drawn up through a capillary tube by means of vacuum, under closely controlled conditions of vacuum and temperature. The viscosity in Pascal-seconds is calculated by multiplying the flow time in seconds with a calibration factor unique to the viscometer tube.

Some findings from the literature pertinent to the scope of work are summarized below:

- In 1994, Zacharias et al. investigated the application of the Brookfield viscometer (ASTM D4402) in South African road grade bitumen specifications and found a good correlation between the Brookfield measurements and the capillary viscometer measurements (ASTM D2171) [1]. However, it was observed that the capillary viscosity values were consistently higher than those from the Brookfield viscometer.

- In 1995, Bahia and Anderson [2] measured the viscosity of a large number of asphalt binders that varied in their source and grade through conventional and new testing methods. The results obtained from the capillary tubes and the rotational viscometer showed that the latter was more suitable to modified binders.

- Li et al. [3] discussed the use of shear rate sweep test to evaluate the viscosity of high viscosity asphalt binders (HVAB) at 60°C and proposed a low shear viscosity at 0.01 s^{-1} as the viscosity evaluating indicator of HVABs. Their findings also indicated that the capillary viscosity of HVABs was 3-8 times higher than zero shear viscosity (ZSV).

- Another study on unmodified and modified binders to evaluate their rheological measurements revealed that for modified binders, the capillary viscometers showed an opposite behavior to that of a Dynamic Shear Rheometer (DSR). The reason was that, when using the capillary viscometers, it is assumed that a full shear flow is developed within the tube, and if it is not fully developed when testing modified binders, extensional thickening characteristics may result and provide misleading readings [4].

2.1.1.4. Advantages and Limitations of Test

Advantages:

- The test is simple and easy to carry out.

- The test itself is fast and requires typically 5 to 6 minutes to carry out.

- The results are ready for use directly and do not require any additional analysis or interpretation by the operator.

Limitations:

- This test method is suitable for use at other temperatures, but the precision is based on 60°C.

- This test method is not suitable for modified/conditioned/recovered asphalt binders due to their non-Newtonian behavior at this temperature. In fact, T202 explicitly warns this at the beginning of the standard, whereas the test is used in Item 300 for certain polymer modified binders.

- Different types of viscometers may result in slightly different measurements (i.e. the viscosity measured in a CMVV (Cannon Manning Vacuum Viscometer) may be from 1 to 5 % lower than either the AIVV (Asphalt Institute Vacuum Viscometer) or MKVV (Modified Koppers Vacuum Viscometer) having the same viscosity range).

- The sample preparation and cleanup take a considerable amount of a technician's time and consumables/solvents.
- The only two parameters obtained from this test are flow time and viscosity. However, the shear rate corresponding to different viscosity measurements cannot be obtained through this test method.

2.1.2. T72 Saybolt Viscosity

2.1.2.1. Other Equivalent or Closely Similar Standards

ASTM D7226

2.1.2.2. Applicable Materials and Test Conditions

- All emulsions (Tables 7, 8, 9, 10, 11) at either 77°F OR 122°F
- Recycling agents (Table 12) at 77°F

2.1.2.3. Utility and Background of Test

According to ASTM D7496 Note 1, this test can be replaced by other tests that are more preferred (T201 and D445), and recommendations are provided to replace the values. Specifically, ASTM D7496 states that *"T201 and ASTM D445 are preferred for the determination of kinematic viscosity. These methods require smaller samples and less time and provide greater accuracy. Kinematic viscosities may be converted to Saybolt viscosities by use of the tables in ASTM D2161. It is recommended that viscosity indexes be calculated from kinematic rather than Saybolt viscosities."*

ASTM D2397 also states that the viscosity of emulsified asphalts can be determined in accordance with either ASTM D7496 (Saybolt viscometer) or ASTM D7226 (Rotational paddle viscometer).

Some findings from the literature pertinent to the scope of work are summarized below:

- Salomon, in his review of new viscosity standards, noted some advantages, such as lower operational costs and smaller equipment footprint for using the rotational paddle viscometer as a replacement for the Saybolt viscometer [8].
- Kee et al. [9] investigated the use of yield stress value to possibly replace the Saybolt grading procedure due to higher consistency in the test results. The preliminary results of their study suggested that the yield stress grading using the slotted plot technique was preferable to other techniques, such as Saybolt viscometer.

- Islam et al. [10] investigated the use of a rotational viscometer in lieu of the Saybolt viscometer and found two ideal test conditions to evaluate the steady-state viscosity of the emulsions. The proposed conditions are listed as 50 rpm at 30 °C, and 30 rpm at 60°C. They also suggested that current specifications for the quality control and quality assurance of low and high viscous emulsions using Saybolt viscometer can be replaced by those of rotational viscometer. Their proposed range is 730 cP and 5–90 cP at 50 rpm and 30 °C for high and low viscous emulsions, respectively.

2.1.2.4. Advantages and Limitations of the Test

In the case of seal coat binders, this test serves as an indicator for the ability to place the material on the surface (shootability).

Advantages:

- The test is simple and easy to perform.
- The duration of the test is short, and measurements are obtained in typically 5 to 6 minutes.
- The test results are direct and do not require any additional analysis or interpretation by the operator.

Limitations:

- The sample preparation and cleanup easily occupy more than 90% of a technician's time (minimum of 1 hr.) to determine the viscosity of a sample.
- Ancillary equipment, such as ovens and water bath, are needed for sample preparation.
- From preparation to application, emulsions experience a history of shear rates. Therefore, it is essential that the viscometer can simulate their behavior at different shear rates. This could be the main drawback of the Saybolt viscometer, which does not have the shear rate adjustments.
- During the Saybolt viscosity test, several time-consuming precautions should be taken to ensure correct measurements. The precautions include checking the level of the bath liquid, the air leakage through the cork, the temperature of the sample outside the viscometer before pouring, etc. [11]
- Saybolt viscosity measurements are not associated with the common viscosity units, which makes it difficult to perform direct comparisons with other viscosity test results.
- From a practical perspective, emulsions transported to a laboratory for quality testing are prone to breaking during the transportation and storage process by the time they are

subjected to a test. This often results in Saybolt numbers that are not representative of the state of the material when it is being used in the field at the time of construction.

2.1.3. T316 Viscosity Determination of Asphalt Binder Using Rotational Viscometer

2.1.3.1. Other Relevant Standards

ASTM D4402

2.1.3.2. Applicable Materials and Test Conditions

PG binders (Table 17) at 135°C

2.1.3.3. Utility and Background of Test

The apparent viscosity of asphalt binder at application temperatures can be determined through this test method. The measured viscosity can be used in further analysis, such as developing temperature viscosity charts for estimating mixing and compaction temperatures. It is also a measure of constructability and whether the binder can be handled properly at the refinery, terminal, or hot mix asphalt facility.

According to Item 300, *"This requirement may be waived at the Department's discretion if the supplier warrants that the asphalt binder can be adequately pumped, mixed, and compacted at temperatures that meet all applicable safety, environmental, and constructability requirements. At test temperatures where the binder is a Newtonian fluid, any suitable standard means of viscosity measurement may be used, including capillary (T 201 or T 202) or rotational viscometry (T 316)."*

2.1.3.4. Advantages and Limitations of Test

Advantages:

- Rotational viscometers are most useful for determining the viscosity of non-Newtonian binders (although specification requirements may not always correctly specify test conditions appropriate for non-Newtonian binders) due to their ability to cover a wide range of shear rates for a single sample.
- Other important binder properties, such as temperature susceptibility and mixing and compaction temperatures, can be determined using a single sample and obtaining viscosity measurements at multiple temperatures.
- The test equipment is not unique to asphalt binder testing, and it is widely available.

Limitations:

- The current PG specifications do not use rotational speeds that accurately reflect the shear rates experienced by the asphalt binder during the mixing and compaction process. This is generally not an issue for unmodified binders, but it predicts artificially inflated mixing and compaction temperatures in the case of modified asphalt binders [12].
- Throughput is constrained by the time to get to temperature, so multiple sets of equipment may be needed for a high production lab.

2.1.4. ASTM D2196 Rheological Properties of Non-Newtonian Materials by Rotational Viscometer

2.1.4.1. Other Equivalent or Closely Similar Standards

ASTM D4402, ASTM D6114, ASTM D7741

2.1.4.2. Applicable Materials and Test Conditions

AR binders (Table 16)

2.1.4.3. Utility and Background of Test

The apparent viscosity, shear thinning, and thixotropic properties of non-Newtonian asphalt binders can be determined through this test method.

ASTM D6114 is exclusively published in specifications for AR binders. This standard requires the viscosity measurements to be performed at 175°C. In addition to the different rotational elements suggested in ASTM D2196, ASTM D6114 allows the use of other rotational viscometers, such as the DSR for determining the apparent viscosity, following the manufacturer's recommendations.

ASTM D6114 provides some guidance with regard to different sizes of rubber materials. For rubber particles larger than mesh No.8, ASTM D7741 is recommended to determine apparent viscosity. For rubber particles smaller than mesh No.50, ASTM D4402 is recommended with the following modifications: (i) stirring the AR binder sample vigorously before starting the test, (ii) acclimating the appropriate spindle in the sample for at least 1 min before testing, and (iii) stirring again immediately before starting spindle rotation. For rubber particles larger than mesh No.50 and smaller than mesh No.8, test method D7741 is recommended as written, and modified D4402 is recommended with the following instructions: the gap between the outer wall of spindle and the inner wall of cup should be at least 5mm.

Some other pertinent information from the literature is summarized below:

- According to many studies, the consistency of rubber-modified asphalt binders during a particular test can be largely affected by the size, shape, and surface area of the rubber particles.

- Leng et al., in their study on the feasibility of using wax-based additives in warm asphalt rubber, used a rotational viscometer to measure the workability of the binders and found substantial impact of the CRM particle characteristics on the viscosity results. Therefore, they recommended not to use rotational viscometers to determine the true workability of asphalt rubber binders, and instead, they suggested further investigation of the viscosity of liquid phase of asphalt rubber as a measure of workability [13].

- Ding et al. [14] used a Brookfield viscometer with different spindle sizes and different rotational speeds to measure the viscosity of AR binders with different crumb rubber contents (0-25%) at 180°C. Their findings suggested that when the rubber content is small, the effect of different sizes of spindles and rotational speeds on the viscosity measurements is relatively low; however, at high rubber contents, the differences between measurements become more significant.

- Lo Presti et al. designed and manufactured a dual helical impeller (DHI) that can be used with a rotational viscometer to have a more realistic viscosity measurement of asphalt rubber. The device was created in such a manner to create a convective-like flow, thus minimizing phase separation. They concluded that in the case of neat bitumen, DHI provides similar results than the standard cylindrical spindle. However, in the case of rubber-modified bitumen, they obtained lower values of apparent viscosity with a more stable trend [15]. In a later study, they developed a computational fluid dynamics (CFD) model to qualitatively compare the viscosity results from DHI and standard spindle, and found that the absence of vertical components of velocity with cylindrical geometry causes the misleading viscosity measurements [16].

- In 2012, Baumgardner and D'Angelo evaluated DSR testing geometry for performance testing of asphalt rubber binders and stated that geometries, such as coaxial cylinder or cup and bob, can better measure the impact of larger particles of crumb rubber modifiers [17].

- Nill and Golalipour [18] also listed a number of advantages for the cup and bob geometry over the parallel plate geometry along with one main disadvantage which is the prolonged thermal equilibrium due to larger sample size.

2.1.4.4. Advantages and Limitations of Test

Advantages:

- The Brookfield rotational viscometer is currently being used by experts as the most common device to measure the viscosity of asphalt binders and its accuracy and reproducibility have been examined extensively compared to other test methods.

Limitations:

- The variability of the results obtained from this test method may be high for asphalt rubber binders with large particles.

2.2. Stiffness Tests

2.2.1. T49 Penetration of Bituminous Materials

2.2.1.1. Other Equivalent or Closely Similar Standards

ASTM D5/D5-M

2.2.1.2. Applicable Materials and Test Conditions

- AC (Tables 2, 3) at 77°F (100 g, 5 sec, unaged)
- Special-use cutbacks (Table 6) 77°F (100 g, 5 sec, unaged)
- Emulsions, Cationic emulsions, Polymer-modified emulsions, Polymer-modified cationic emulsions, (Tables 7, 8, 9, 10) 77°F (100 g, 5 sec, unaged)
- Crack sealer (Table 14) at 77°F (100 g, 5 sec, unaged) and Rubber crack sealers (Table 15) at 77°F (150 g, 5 sec, unaged), 32°F (200 g, 60 sec, unaged)
- AR binder (Table 16) at 77°F (100 g, 5 sec, unaged) and 39.2°F (200 g, 60 sec, aged & TFOT-aged)

2.2.1.3. Utility and Background of Test

Generally, the penetration test is used to quantify the fluidity or consistency of asphalt binders by releasing a needle of precise weight into a cup of binder and measuring the needle's depth of penetration.

According to Asphalt Institute, "While this system is no longer commonly used in the United States, the penetration test is still a valuable indicator of consistency of source or process."[101]

Some findings from the literature pertinent to the scope of work are summarized below:

- The results from the penetration test are highly empirical and most of the previous efforts to express these measurements in fundamental units have been unsuccessful [22].

- The penetration index obtained from this test method is still being used in many countries as a quality control measure and a temperature susceptibility parameter. However, several publications have appeared in recent years documenting some concerns regarding the validity of this parameter [23].

- Sybilski [24] studied the relationship between three binder properties (penetration depth, softening point, and zero-shear viscosity (ZSV) at 60°C) and the rutting resistance N_{10} parameter. First, a simple regression analysis was performed on each binder property, and the results indicated poor correlation for the penetration depth and the softening point, while a strong correlation for the ZSV. Next, a multiple regression analysis was conducted with the three binder properties as independent variables, and the results showed much better statistical correlation than those of simple regression.

- Davis [25] discussed penetration as it relates to stiffness or viscosity of asphalt and developed a method to calculate an equivalent viscosity from penetration. This calculation gives the viscosity of an asphalt binder at the temperature at which the penetration test is performed. This led to development of Tex-535-C, "Calculating Viscosity from Penetration" [26], which can be used to suggest DSR limits for replacing penetration with DSR requirements.

2.2.1.4. Advantages and Limitations of Test

In the context of binders used for seal coats, the penetration test results also serve as an indicator of flushing resistance.

Advantages:

- The test is simple, easy to conduct, fast, and requires low capital cost in terms of equipment.

- The results produced by the test are directly used and no additional data analysis or interpretation is required to be performed by the operator.

- The test is mainly performed at 25°C (77°F), which is close to the average pavement service temperature.

Limitations:

- The only two results obtained from this test are the penetration depth and the penetration index, both of which fail to be expressed in fundamental units.

- The penetration index parameter obtained from this test is calculated based on the changes in binder properties over a relatively small range of temperatures, and therefore, these results may be misleading if extrapolated to temperature extremes.

- Slight variations while performing this test can cause large differences in the results. Errors are usually due to: (i) sampling and sample preparation, (ii) variations in temperature, and (iii) condition of the apparatus (straightness of the needle, cleanliness, etc.) [27].

2.2.2. T53 Softening Point

2.2.2.1. Other Equivalent or Closely Similar Standards

ASTM D36

2.2.2.2. Applicable Materials and Test Conditions

- Polymer-modified AC (Table 3)
- Polymer-modified cationic emulsions (Table 10)
- Polymer-modified emulsion crack sealers (Table 14)
- Rubber crack sealer (Table 15)
- AR binder (Table 16)

2.2.2.3. Utility and Background of Test

This test is primarily used as a consistency check for modified asphalt binders. Two samples of bitumen cast in shouldered brass rings are heated at a prescribed rate in a liquid bath. The softening point is reported as the mean of the temperatures at which the two samples soften enough to fall a distance of 25 mm through a hole that is plugged using the bitumen sample.

Asphalt binders with higher softening points are preferred in warmer places, and those with lower softening points are preferred in colder climates.

Some findings from the literature pertinent to the scope of work are summarized below:

- According to Bahia and Anderson [2], softening point is an empirical single-point measurement which cannot be expressed in engineering units and cannot be directly related to the rheology of asphalt binders.

- Sybilski [24] studied the relationship between three binder properties (penetration depth, softening point, and zero-shear viscosity (ZSV) at 60°C) and the rutting resistance N_{10} parameter. First, a simple regression analysis was performed on each binder property, and the results indicated poor correlation for the penetration depth and the softening point, while a strong correlation for the ZSV. Next, a multiple regression analysis was conducted with the three binder properties as independent variables, and the results showed much better statistical correlation than those of simple regression.

- This test method is useful for testing materials to be used as joint and crack sealers, because it serves as a surrogate indicator for the resistance of the material to bleeding and tracking.

2.2.2.4. Advantages and Limitations of Test

General:

- In the case of crack seal materials, this test also serves as an indicator of tracking potential.
- In the case of seal coat binders, this test also serves as an indicator of flushing resistance during summer.

Advantages:

- The test is simple and easy to perform.
- The results from this test method are directly available and do not need any additional analysis or interpretation by the operator.
- This test method can serve as an indicator for the efficiency of crack-sealers to prevent cracking.

Limitations:

- Empirical measurements, such as softening point, use different loading modes, different loading rates, and sometimes different temperatures, which makes it difficult to combine and relate them to the fundamental rheological properties [2].
- Performing this test, as well as the sample preparation and the cleanup procedures, consume a significant amount of an operator's time and attention.
- This test is very sensitive to extraneous factors, such as vibrations, rate of heating, and the leveling of the instrument.

2.3. Ductility and Elastic Recovery Tests

2.3.1. T51 Ductility of Asphalt Materials

2.3.1.1. Other Equivalent or Closely Similar Standards

ASTM D113/ASTM D6084/Tex-539-C

2.3.1.2. Applicable Materials and Test Conditions

- AC (Table 2), RC & MC cutback (Tables 4 and 5), Emulsified and Cationic Emulsified asphalt (Tables 7 and 8), Polymer Modified (PM) emulsified asphalt (Table 10) at 77°F, 5 cm/min
- PM AC (Table 3), Special-use cutback asphalt (Table 6), PM Emulsified asphalt (Table 9) at 39.2°F, 5 cm/min
- PG binders (Table 17)

2.3.1.3. Utility and Background of Test

This test method measures the ductility of asphalt materials by the distance to which they will elongate before breaking when the two ends of the material are pulled apart at a uniform speed. The elastic recovery of PG binders can also be assessed through this test.

Ductility tests at 77°F (25°C) are primarily performed to identify the cohesion of the asphalt binder, while those at 39.2°F (4°C) are performed to measure binder modification.

Some findings from the literature pertinent to the scope of work are summarized below:

- The conventional ductility test at low temperatures is still being used in some specifications as a performance indicator for asphalt modification. A review of the literature suggests that some correlation exists between the binder ductility and the cracking behavior of the pavements, provided that the measurements are obtained at the appropriate temperature.
- In 1958, in an attempt to find correlations between binder characteristics and the actual pavement behavior, Doyle tested the ductility of binders at two temperatures, 55°F and 77°F, and found a fairly good correlation with the pavement performance at 55°F, but no correlation at 77°F [29] .
- In 1976, Anderson et al. found some correlation between force ductility and transverse cracking [30].
- In 1984, Kandhal and Koehler evaluated those properties of asphalt binders that are associated with aging and their relationship to pavement performance. Their study suggested that among penetration at 77°F, viscosity at 140°F, and ductility at 60°F, only the ductility appeared to be consistent with pavement performance after ten years [31].
- In a study performed on the reliability of the ductility test for modified binders, Tabatabaee et al. [32] reported no fundamental relationship between ductility and cracking damage, nor with the percent recovery and J_{nr} values of asphalt binders. To address these issues, the authors proposed a specialized DSR procedure designed to

measure the true ductility of modified binders at 39.2°F. It should be noted that for neat asphalt binders, the results from the conventional and the DSR ductility tests matched up relatively well.

2.3.1.4. Advantages and Limitations of Test

Advantages:

- Lack of sophistication, easy operation, and fast measurement are the major advantages of this test method.
- The results from this test method need no interpretation by the operator.

Limitations:

- Performing this test, as well as the sample preparation and the cleanup procedure, involve a significant amount of an operator's time.
- The amount to which asphalt binder extends during the ductility test is a function of the interaction between the test temperature, temperature susceptibility, pulling rate and sample geometry, and perhaps other parameters. Therefore, unless the main effect of each of the influencing factors is not independently evaluated, the test results may be misleading [32], [33].

2.4. Integrity Tests

2.4.1. D44-14 Solubility in TCE Test

2.4.1.1. Other Equivalent or Closely Similar Standards

ASTM Designation: Previously D5546 (using Toluene); now replaced with D2042

2.4.1.2. Applicable Materials and Test Conditions

- Asphalt Cement (Table 2)
- Rapid-Curing (Table 4), Medium Curing (Table 5) and Special-Use Cutback Asphalts (Table 6): Tested on T78 distillation residue
- Emulsified Asphalt (Table 7), Cationic Emulsified Asphalt (Table 8) Polymer-Modified Emulsified Asphalts (Table 9), Polymer-Modified Cationic Emulsified Asphalt (Table 10), and Specialty Emulsions (Table 11): Tested on T59 distillation residue

2.4.1.3. Utility and Background of Test

Two grams of test material are first dissolved in 100 ml of Type I trichloroethylene (TCE) or 1,1,1 trichloroethane. The solution is then filtered through a glass microfiber filer pad (32, 35 or 37 mm), leaving behind insoluble particles that did not pass through the filter paper. These particles are washed, dried, and weighed until they achieve constant weight.

Some findings from the literature pertinent to the scope of work are summarized below:

- Previous studies have indicated that the solvent n-Propyl Bromide might serve as a safer alternative to trichloroethylene [38].
- Asphalt Institute [2] conducted a survey about the use of this test for asphalt binders. The survey included questions about the types of binders that were tested for solubility, the frequency of such tests, the standards followed, and the types of solvents used (88% TCE, 10% Toluene, 2% Others). Some pertinent comments were:
    - "We have been concerned that makers of tire-rubber modified binders like AC-15-5TR have proposed solubility as a way to scare off competition."
    - "It is an artifact that no longer serves a purpose and the users simply need to be educated to that fact so they can remove the test from the specs."
- Bowers et al. [3] investigated sequential dissolution of asphalt binder in TCE, decahydronaphthalene (decalin), tetrahydrofuran (THF), and toluene, and found toluene not as effective as the other three.

2.4.1.4. Advantages and Limitations of Test

General:

In a broad sense, this test is used to detect contamination of asphalt binder with excess amount of extraneous, insoluble components or particulates.

Advantages:

- The sample preparation method is easy to follow (prepare a solution) and execute (filter, heat, and weigh).
- The parameters measured from this test are straightforward, with higher % insoluble or lower % solubility as a sign of possible contamination or non-asphalt materials in the binder.
- The test does not require a substantial amount of time to conduct.
- The capital cost of the equipment is low (approx. $100).

Limitations:

- The T59 test for cutback asphalts or T78 test for emulsified asphalts must be run before this test.
- Trichloroethylene and 1,1,1 trichloroethane in the presence of heat and moisture can form acids, which are extremely harmful to health if inhaled. It would be better to use a relatively safer solvent (such as Toluene) or an analytical method (such as FTIR) to detect the presence of impurities in asphalt binder.

Similar Tests:

AASHTO T111 ash test [39], Sequential approach [39], [40]

2.4.2. Tex-509-C Spot Test of Asphaltic Materials

2.4.2.1. Other Equivalent or Closely Similar Standards

AASHTO T102

2.4.2.2. Applicable Materials and Test Conditions

- AC (Table 2)
- Rapid curing and medium curing cutback asphalts (Tables 4, 5)

2.4.2.3. Utility and Background of Test

According to the AASHTO T102, the spot test is used to detect overheating of asphaltic materials during processing, as well as to indicate the compatibility of the components of an asphalt binder. A "negative" result indicates no overheating and good compatibility, while a "positive" result indicates some level of damage due to overheating and poor compatibility. Tex-509-C is similar to AASHTO T102 with note for use of "In lieu of the standard naphtha, perform all testing using a blend of 65% heptane and 35% xylene."

This test is performed on the original form of AC binders and on the cutback residue from the distillation test. Some other pertinent information from the literature is summarized below:

- According to the literature, due to the recent advancements in the refining practices that prevents "breaking" of asphalt binders, the spot test is no longer being performed on a routine basis.

2.4.2.4. Advantages and Limitations of Test

Advantages:

- Simple procedure and easy operation are the major advantages of this test method.
- The results from this test method need no interpretation by the operator.

Limitations:

- The sample preparation and cleanup easily occupy more than 90% of a technician's time (minimum of 1 hr.).

2.5. Safety Tests

2.5.1. AASHTO T48 "Flash Point of Asphalt Binder by Cleveland Open Cup" and T79 "Flash Point with Tag Open-Cup Apparatus for Use with Material Having a Flash Point Less Than 93°C (200°F)"

2.5.1.1. Other Equivalent or Closely Similar Standards

ASTM D92, ASTM D1310

2.5.1.2. Applicable Materials and Test Conditions

T48

- AC and Polymer-modified AC (Tables 2, 3)
- Polymer-modified cationic emulsions and specialty emulsion (Tables 10, 11)
- Recycling agents and emulsified recycling agents (Table 12)
- Rubber-Asphalt crack sealer (Table 15)
- AR binders (Table 16)
- PG binders (Table 17)

T79

- Rapid curing, medium-curing, and special-use cutback asphalt (Tables 4, 5, 6)

2.5.1.3. Utility and Background of Test

Flash point is defined as the lowest temperature of the test specimen at which application of the test flame causes the vapors of the specimen to ignite. Both AASHTO T48 and T79 standards are used to determine the flash point of asphalt binders for safety purposes. The main difference between them is that the Cleveland open cup tester (T48) is used to determine the flash point of

asphalt binders within the range of 219°C (426°F) and 400°C (750°F), while the Tag open cup tester (T79) is used for binders having flash points below 93°C (200°F). The test cup in T48 is made of brass or another metal of similar conductivity, while in T79, it is made of molded clear glass. Some features of these two testers and test conditions are compared in Table 2.1 below.

Table 2.1 Flash Point Test Summary.

Test Method	Test cup outside diameter (mm)	Test cup depth (mm)	Bath media	Rate of increase in temperature (°C/min)
T48	67.5-69.0	32.5-34.0	-	Initially 10-20, Then 4-10
T79	Upper part: 55.1±1.9 Lower part: 50.8±1.6	47.6±1.6	Water/ water glycol solution	1.0±0.3

Some other pertinent information from the literature is summarized below:

- ASTM D7094 states "Flash point by modified continuously closed cup tester" is intended to determine the flash point of fuels including diesel/biodiesel blends, lube oils, solvents, and other liquids. It is suitable for materials having a flash point from 35°C to 225°C. The closed cup procedure in this test method prevents the escaping of vapors; therefore, the measured flash point is usually a few degrees lower than that of an open cup, which eventually ensures a safer practice. This test method can be a potential replacement for T48 and T74 due to some benefits, such as safer procedures and a smaller amount of material to be tested. It is worth noting that in December 2013, this test method was officially accepted as an alternative method for measuring the flash point of fuel oils, diesel fuel oils, gas turbine fuel oils, and Kerosene.

- It is stated in ASTM D7094 that the flash point values are not a constant material property and could be a function of some factors, such as the apparatus design, the operational procedures carried out, and the condition of the apparatus used. Therefore, it is essential to specify which standard test method was used for measuring the flash point of a specimen.

- Another continuously closed cup flash point tester, ERAFLASH, produced by Eralytics, has been recently used by researchers in studies on engine oil [41]. This technology can cover a temperature range of -25°C to 420°C and is equipped with a significantly faster cooling down system compared to other closed cup testers. The ERAFLASH apparatus also features a contamination prevention technology which minimizes any required cleaning.

2.5.1.4. Advantages and Limitations of Test

Advantages:

- Compared to other standard test methods, AASHTO T48 and T79 have more widely been practiced in the asphalt industry.

Limitations:

- Compared to the closed cup methods, the results from the open cup methods are more likely to be influenced by outside elements in the laboratory, such as the ambient pressure.

2.6. Emulsion Tests

2.6.1. T 50 Float Test

2.6.1.1. Other Equivalent or Closely Similar Standards

ASTM D139-16 except with two new additions:

- One new section immediately before Section 2.1, which replaces the references for ASTM D244 and ASTM D6997 with the reference to AASHTO T59 (distillation test) alone, and
- Two new sentences at the end of Section 7.3, which provide an option for pouring the T59 distillation residue directly into a container and maintaining it at the "proper temperature" for use in float test. (Note: Section 7.3 also includes a reference to Section 26 of ASTM D244, which essentially recommends the pouring the distillation residue into the collar at or near 260°C; AASHTO T50 does not specify exact value of "proper" pouring temperature.)

2.6.1.2. Applicable Materials and Test Conditions

Applicable Materials: T59 distillation residue of

- Emulsified Asphalt (Table 7): Min. 1200 sec. for HFRS-2 and AES-300
- Polymer-Modified (PM) Emulsified Asphalt (Table 9): Min. 1200 sec. for HFRS-2P, AES-150P, AES-300P, and AES-300S
- Polymer-Modified Cationic (PMC) Emulsified Asphalt (Table 10): Min. 1800 sec. for CHFRS-2P
- Specialty Emulsions (Table11): 50-200 sec. for AE-P

Test Conditions:

- Emulsified Asphalt, PM Emulsified Asphalt, PMC Emulsified Asphalt: Conditioned in water bath at 5°C; Tested in a water bath at 60°C (140°F)
- Specialty Emulsions: Conditioned in water bath at 5°C; Tested in a water bath at 50°C (122°F)

2.6.1.3. Utility and Background of Test

Asphalt emulsion is poured into a brass collar, forming a plug at its center. The collar is conditioned at 5°C in a water bath for 15 to 30 min and then screwed into an aluminum float at its center. The float-and-collar assembly is again conditioned at 5°C in the same water bath for one minute and then placed another water bath at 60°F or 50°F as dictated by the binder type. The time taken by water to break through the plug after the binder gets softer in this water bath is recorded as the float value for the binder.

The Float Test is used for two applications. The first application is for materials that are very fluid and possibly too fluid to measure viscosity conveniently (AE-P is the only material in this category). Otherwise, it is used to measure the High Float (HF) gel structure introduced by the high float property that is exclusive to emulsions using these emulsifiers. These HF emulsions have been anionic in the past, but later CHFRS-2P was also included in the materials relevant for this test. It is unclear whether this property is an artifact of distillation of the emulsion. Some industry experts suggest that this property can be developed while curing on the pavement surface, but it occurs over a longer period of time.

Some findings from the literature pertinent to the scope of work are summarized below:

- Suda [42] recently used the float and DSR tests to investigate rheological properties of one regular PG 58-28 asphalt and two asphalt emulsion distillation residues. Based on these tests, the researcher determined that, unlike regular asphalt, the high-float asphalt emulsions are non-linearly elastic in nature, and exhibit yield stress during stress-ramp tests. The researcher concluded this test and parameter can potentially replace the float test.

2.6.1.4. Advantages and Limitations of Test

General:

The test serves as measure of consistency [42] or resistance to bleeding (flow) at an elevated temperature through the formation of a gel-structure.

Advantages:

- The test method is straightforward, and easy to follow and execute.
- The measured parameters are obtained directly (read the time from a stopwatch) and are straightforward to interpret (%longer time ➔ high float).
- The capital cost of equipment associated with this test is low (float + collar= approx. $200).

Limitations:

- T59 distillation residue is needed to prepare the test sample.
- The measured parameter (i.e., time) is not a mechanical, but an empirical indicator.
- Pouring temperature is not well defined.
- Chances of errors due to variations in temperature: The 1-minute-long conditioning of binder at 5°C may not be enough to compensate the drop in temperature that happens while attaching the collar into the float.

2.6.2. T59 "Standard Method of Test for Emulsified Asphalts-Recovery by Distillation and Evaporation"

2.6.2.1. Other Equivalent or Closely Similar Standards

2.6.2.1.1. Distillation:

- ASTM D6997
- ASTM D7403

2.6.2.1.2. Evaporation:

- AASHTO PP72
- ASTM D6943

2.6.2.2. Applicable Materials and Test Conditions

2.6.2.2.1. Distillation:

- Emulsified asphalt, cationic emulsified asphalt, PM emulsified asphalt, PM cationic emulsified asphalt, and Specialty emulsions (Tables 7, 8, 9, 10, 11)

2.6.2.2.2. Evaporation:

- Specialty emulsions (Table 11)
- Recycling agent and emulsified recycling agent (Table 12)
- PM asphalt-emulsion crack sealer (Table 14)

2.6.2.3. Utility and Background of Test

Both distillation method and evaporation method can be used for quantitative determination of residue in an emulsified asphalt. The distillation method can also determine the oil distillate in the emulsion.

In the distillation method, 200 gram of an emulsion is poured into the distillation pot and heat is applied until the completion of distillation. The total distillation must be completed in 60±15 min from the first application of heat. Immediately after that, the mass of the distillation pot and accessories is determined, and the percentage of residue is recorded. The percentage of oil distillate is also calculated by recording the volume of the condensed vapors collected in a graduated cylinder. If further testing is needed, the residue is poured into any suitable mold or container.

In the evaporation method, 200 g of an emulsion is poured into four beakers (50 g/beaker), and the beakers are placed in an oven for two hours at a temperature of 163°C. The beakers are then removed from the oven; the residue is stirred thoroughly; and the beakers are replaced in the oven for an additional one hour. After that, the beakers are removed from the oven and allowed to cool at room temperature. The mass of the beakers is determined, and the percentage of residue for each beaker is calculated. The average of these four values is the percentage of residue by evaporation for the emulsion sample.

T59 states that "The method for residue by evaporation tends to give an asphalt residue lower in penetration and ductility than the distillation method." It also states that "If residue by evaporation fails to meet the requirements for properties specified for residue by distillation, the tests shall be repeated using the distillation method."

Some findings from the literature pertinent to the scope of work are summarized below:

- The AASHTO PP72 standard covers two methods (A and B) for recovering the residue from emulsions using low-temperature evaporative techniques. In section 3 of this standard, it is stated: "Use this practice in place of recovery techniques such as those in T 59; the temperatures used in that standard may negatively affect the residue." In general, compared to T59, this test method uses lower temperatures and longer testing periods to provide conditions that are very close to that of application techniques for these materials. However, as reported by Reinke in a TRB workshop in 2013 [43], procedure A was not

suitable to a production environment due to the extended time of testing (>48 h), and Procedure B was found to result in residues that were substantially stiffer than the base binder from which the emulsion had been produced.

- The ASTM D7403 standard determines the percentage of residue in emulsions by applying a minimum vacuum of 88 kPa at a temperature of 135°C in 60 minutes. This test method is not recommended to be used for buy/sell purposes until a precision statement is available for it.

- The ASTM D7404 standard uses a moisture analyzer balance (MAB) to rapidly determine the percentage of residue in emulsified asphalts and is applicable to all nonsolvent-containing emulsion types. This method requires a minimum of 1 g and up to 3 g of emulsified asphalt. The sample of emulsion is placed in an open sample tray and heated to a preset temperature, which in no case exceeds 163°C, and the residue is automatically calculated by the instrument. A good correlation is found between this method and the ASTM D6943 method [43].

- The major issue with the T59 test method is the high temperature involved in the procedure which could lead to degradation of the polymer network in the emulsion. This problem was addressed by the ASTM D7497 standard which is equivalent to AASHTO PP72 procedure A. Studies conducted on strain tolerance, elastic recovery, and total accumulated strain showed that this method does not degrade the polymer [44], [45].

- In recent years, the use of vacuum to accelerate the recovery of emulsions at lower temperatures has been investigated. In a TRB workshop on progress towards performance-graded emulsified asphalt specification [43], Reinke suggested replacing the forced draft oven with a vacuum oven and reducing the testing time from the 6 hr. required by AASHTO PP72 to 2 hr. without negatively impacting the residue. This approach was later evaluated by researchers and appeared feasible. For instance, Islam et al. [46] proposed a method called vacuum drying in which the pressure inside the oven is lowered to a point that oxidation is minimized, and the residue is cured enough after 3 hr. at 60°C.

- In the TRB workshop, Salomon proposed an extension to the ASTM D7404, using a MAB to recover the residue. In his method called MAB-DSR, the recovery is performed in the DSR silicon mold, and directly placed on the parallel plates for further testing [43].

2.6.2.4. Advantages and Limitations of Test

Advantages:

- The results produced by the test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- In the case of polymer modified emulsions, the high temperature involved in the T59 procedure may cause a degradation in the polymer network.
- One major limitation of the T59 evaporation procedure is that the sample of emulsion is held in an open beaker for 2 hr. at a high temperature, and this causes oxidative aging for the emulsion residue.

2.6.3. T59 "Standard Method of Test for Emulsified Asphalts- Sieve Test"

2.6.3.1. Other Equivalent or Closely Similar Standards

ASTM D6933, ASTM D244

2.6.3.2. Applicable Materials and Test Conditions

- Emulsified asphalt, cationic emulsified asphalt, PM emulsified asphalt, PM cationic emulsified asphalt, and Specialty emulsions (Tables 7, 8, 9, 10, 11)
- Recycling agent and emulsified recycling agent (Table 12)
- PM asphalt-emulsion crack sealer (Table 14)

2.6.3.3. Utility and Background of Test

This test is used to verify that the emulsions do not contain excessive amount of particles remaining on a No. 20 sieve, which may cause problems in handling and application of the materials.

The first step in this test is to determine the appropriate testing temperature based on the Saybolt viscosity. This can be done by review of the specification limits for an emulsion and not based on performing the test. 1000 g of the emulsion is poured through a sieve and washed with distilled water. The sieve is placed on a pan, and the assembly is heated in an oven for 2 hr. at 163°C. The assembly is then cooled in a desiccator, and the percentage of the residue retained on the sieve is reported.

2.6.3.4. Advantages and Limitations of Test

Advantages:

- This test is simple, easy to conduct, fast, and requires low capital cost in terms of equipment.

- The results produced by this test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- Performing the test results in test equipment that must be cleaned, generally with solvents.

2.6.4. T59 "Standard Method of Test for Emulsified Asphalts-Miscibility"

2.6.4.1. Other Equivalent or Closely Similar Standards

ASTM D244, ASTM D6999

2.6.4.2. Applicable Materials and Test Conditions

- Emulsified asphalt, PM emulsified asphalt, and Specialty emulsions (Tables 7, 9, 11)
- Recycling agent and emulsified recycling agent (Table 12)

2.6.4.3. Utility and Background of Test

This test method is applicable to Medium Setting (MS) and Slow Setting (SS) emulsions. 50 mL of emulsion is gradually diluted with 150 mL distilled water at a testing temperature of 21°C to 25°C, and allowed to stand for 2 hr. The mixture is then examined for the presence of any coagulation or separation of the asphalt which are an indication of breaking. If no breaking is observed, the emulsion passes the test.

ASTM D6999 specifies a temperature of 25±3°C for both the emulsion sample and the water to be added. This standard also states that prior to the test, emulsions with viscosity testing requirements of 50°C (122°F) shall be heated to 50±3°C to achieve homogeneity.

Table 11 of Item 300 specifies that the specialty emulsions shall be diluted with 350 mL of distilled or deionized water. Table 12 requires a calcium chloride solution in place of water for testing the miscibility of emulsified recycling agents.

2.6.4.4. Advantages and Limitations of Test

Advantages:

- This test is simple, easy to conduct, fast, and requires low capital cost in terms of equipment.

- The results produced by this test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- The test relies on subjective evaluation of damage to the emulsion.

2.6.5. T59 "Standard Method of Test for Emulsified Asphalts- Demulsibility & Cement Mixing"

2.6.5.1. Other Equivalent or Closely Similar Standards

- Demulsibility
  ASTM D6936
- Cement mixing
  ASTM D6935

2.6.5.2. Applicable Materials and Test Conditions

- Demulsibility
  Emulsified asphalt, cationic emulsified asphalt, PM emulsified asphalt, PM cationic emulsified asphalt, and Specialty emulsions (Tables 7, 8, 9, 10, 11)
- Cement mixing
  Emulsified asphalt, cationic emulsified asphalt (Tables 7, 8)

2.6.5.3. Utility and Background of Test

Demulsibility

The stability in terms of chemical breaking of Rapid Setting (RS) and MS anionic and cationic emulsions can be measured through the demulsibility test method. The amount of available asphalt that is broken from the emulsion is measured, and the type of emulsion RS or MS is identified. For anionic and cationic emulsions, the solutions used to break the emulsion are calcium chloride and dioctyl sodium sulfosuccinate, respectively.

100 g of the emulsion residue is poured into a beaker, and the appropriate solution is added within approximately 2 min. Then, the contents are stirred continuously and vigorously to ensure thorough mixing of the solution and the emulsion. The mixture is decanted on a wire cloth and placed in a beaker with a metal rod. The assembly is placed in an oven for 1 hr. at 163°C (325°F), then removed and allowed to cool, and the demulsibility is calculated as the percentage of the mass of demulsibility residue over the mass of the emulsion residue.

Cement Mixing

The stability of a SS or CSS emulsion in terms of their ability to mix with a finely divided, high surface area material without breaking can be measured through this test method. The emulsion is diluted with distilled water to a residue of 55%, as determined by distillation or evaporation for 3 hr. at 163°C. 100 mL of the diluted emulsion is added to 50 g of a cement passing the No.80 sieve, and the mixture is stirred at once with a steel rod for 1 min. Then, 150 mL distilled water is added, and stirring is continued for 3 min. The mixture is poured through a No.14 sieve and placed in the oven at 163°C. The mass of the residue is determined and reported as the percentage of break in the cement mixing test.

2.6.5.3.1. Literature on Demulsibility and Cement Mixing

Some findings from the literature pertinent to the scope of work are summarized below:

- To characterize the stability of RS emulsions, Banerjee et al. [47] developed a simple quantitative test method based on electrokinetic techniques. This method determines the rate at which an asphalt emulsion breaks when subjected to an electric field and differentiates between formulations of the same emulsions at different dilution rates. The test can record the current flow in real time and has shown its potential as an alternative to the conventional test methods.

- Kadrmas, in his paper published in a TRB circular in 2006, stated that even though the cement mixing test is an important laboratory test for emulsions, especially in cold mix applications, the actual stability in the field needs to be verified with the materials to be used [48].

2.6.5.4. Advantages and Limitations of Test

Advantages:

- These tests are simple, easy to conduct, and require low capital cost in terms of equipment.

- The results produced by these tests are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- Due to the difficulties associated with the breaking and sieving of the materials, these tests are not typically performed in the field [47].

2.6.6. T59 "Standard Method of Test for Emulsified Asphalts- Coating Ability and Water Resistance"

2.6.6.1. Other Equivalent or Closely Similar Standards

ASTM D244

2.6.6.2. Applicable Materials and Test Conditions

- Emulsified asphalt, cationic emulsified asphalt, PM emulsified asphalt (Table 7, 8, 9)

2.6.6.3. Utility and Background of Test

This test method covers the determination of the ability of an asphalt emulsion (MS or SS) to (*1*) coat an aggregate thoroughly, (*2*) withstand a mixing action while remaining as a film on the aggregate, and (*3*) resist the washing action of water after completion of the mixing. The aggregate used in this test is typically coarse-graded calcareous; however, if other types of aggregates are used, laboratory washing, air drying, and mixing the aggregates with calcium carbonate shall be omitted throughout the test.

The test is conducted at 75±10°F. There are two procedures available in the standard: (i) test with dry aggregate, and (ii) test with wet aggregate. 4.0 g of $CaCO_3$ is added to 461 g of the air-dried aggregate and mixed for 1 min to obtain a uniform film of the Ca CO_3 dust on the aggregate. 35 g of emulsion is added to the mix and vigorously mixed for 5 min. At the end of mixing period, the excess emulsion is drained by slightly tilting the pan. Approximately one half of the mixture is removed and placed on an absorbent paper to evaluate the coating. The coating ability is recorded as "good," "fair," and "poor," where good is an indication of fully coated aggregates, fair indicates a condition of more area of coated than uncoated, and poor is an indication of more uncoated area than coated. In the next step, the other half of the mixture remaining in the pan is sprayed with water; water is poured off; and the procedure is repeated until the overflow water runs clear. Water is drained from the pan, and the aggregates are transferred to an absorbent paper to visually determine the coating ability. In the final step, the evaluation is repeated after the mixture has been dried in the laboratory at room temperature.

Some findings from the literature pertinent to the scope of work are summarized below:

- Swiertz et al. [49] in their study to evaluate the laboratory coating of cold mix asphalts used an image analysis technique as a more objective and reliable method than visual observation. This technique can differentiate between coated and uncoated particles based on the contrast of pixel intensities. This method was further refined in a later study. The authors developed quantitative models to predict the level of aggregate coating more precisely by isolating the most significant factors influencing the coating ability, such as emulsion content, aggregate moisture, and aggregate gradation. Overall, this procedure

was found reliable and could introduce performance-based limits on cold mix asphalts in terms of aggregate coating [50].

2.6.6.4. Advantages and Limitations of Test

Advantages:

- The results produced by the test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- The visual estimation of coated/uncoated area can be highly subjective if multiple operators perform the test.

2.6.7. T59 "Standard Method of Test for Emulsified Asphalts- Storage Stability"

2.6.7.1. Other Equivalent or Closely Similar Standards

- ASTM D244, ASTM D6930

2.6.7.2. Applicable Materials and Test Conditions

- Emulsified asphalt, cationic emulsified asphalt, PM emulsified asphalt, PM cationic emulsified asphalt, and Specialty emulsions (Tables 7, 8, 9, 10, 11)
- PM asphalt-emulsion crack sealer (Table 14)

2.6.7.3. Utility and Background of Test

This test method determines the ability of emulsions to remain as a uniform dispersion during storage. It is applicable to all types of emulsions listed in Item 300, except MS polymer-modified cationic emulsions and SS specialty emulsions.

The T59 is similar, but not identical to the ASTM D6930 procedure. Both tests require the emulsion to be placed in undisturbed simulated storage for one day prior to testing. The difference in percent residue between two samples of the emulsion – one from the top and one from the bottom – is determined and reported in percentage as the storage stability. Both tests use the evaporation method to recover the residue. The slight variations between the two tests are listed below:

- T59 does not specify any heating and stirring of the emulsion before testing to achieve homogeneity. ASTM D6930, however, has two requirements based on the Saybolt viscosity testing temperatures: (1) emulsions with viscosity testing requirements of 50°C

(122°F), shall be heated to 50±3°C in their original container and stirred to ensure homogeneity, and (2) emulsions with viscosity testing requirements of 25°C (77°F), shall be stirred or mixed in their original container to achieve homogeneity.

- T59 uses two 500-mL cylinders and tests two representative samples from the emulsion; whereas, ASTM D6930 uses only one cylinder. The storage stability result reported from T59 is the average of the two individual cylinder results.

Some findings from the literature pertinent to the scope of work are summarized below:

- The stability of asphalt emulsions can be evaluated by various techniques, such as rheology tests, particle size measurements, and optical microscopy.

- Zhai et al. [51] used a temperature sweep test to predict the storage stability for different emulsions. Changes in the G* and phase angle of multiple emulsions were measured, and the differences in the crossover temperatures (δ=45°) were used to explain the relative rate of breaking. Overall, they concluded that this method can be used as a useful tool to predict the stability of asphalt emulsions.

- Hou et al. [52] evaluated the stability of four different asphalt emulsions using the storage stability test and laser particle size analyzer (LPSA) and found a linear relationship between the second power of particle size and the storage stability of asphalt emulsions. They also used a differential scanning calorimetry (DSC) to evaluate the thermal stability and temperature susceptibility of asphalt emulsions through measuring their enthalpy change, and found no consistent ranking between storage stability and thermal stability.

- Wang et al. [53] also used a laser diffraction technique to study the variation in D90 (droplet diameter for the 90^{th} cumulative mass percentile) with respect to temperature and storage time, and found this method promising to provide a fast stability prediction method.

2.6.7.4. Advantages and Limitations of Test

Advantages:

- This test is simple, easy to conduct, fast, and requires low capital cost in terms of equipment.

- The results produced by this test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- According to the standard, the only function of this test is to measure the permanence of the dispersion as related to time, and it shall not be used as a measure of other stability aspects involved in use. If rheological tests, such as temperature sweep, can be used as an alternative, the emulsions can not only be characterized for their workability, but also for their performance.

2.6.8. T59 "Standard Method of Test for Emulsified Asphalts – Freezing Test"

2.6.8.1. Other Equivalent or Closely Similar Standards

ASTM D6929

2.6.8.2. Applicable Materials and Test Conditions

- Emulsified asphalt (Table 7)

2.6.8.3. Utility and Background of Test

This test method evaluates the homogeneity of asphalt emulsions that are desired to be used, stored, or transported under less-than-ideal weather conditions. Item 300 specifies that this test applies only when the engineer designates material for winter use.

Approximately 400 g of the emulsion is placed in a container and exposed to an air temperature of -18±5°C for a minimum of 12 hr. (ASTM D6929: 12 to 18 hr.). At the end of freezing period, the emulsion is placed in the ambient temperature to thaw, and the freeze-thaw procedure is repeated for two other cycles. After the third cycle, the emulsion is either "homogenous" or "broken" if the separated distinct layers cannot be rendered homogeneous by stirring at ambient temperature.

2.6.8.4. Advantages and Limitations of Test

Advantages:

- The results produced by the test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- Each freeze-thaw cycle in this test requires at least 12 hr. to complete. Therefore, a total of at least 36 hr. is needed to complete this test, which consumes a significant amount of an operator's time and attention.

2.6.9. T59 “Standard Method of Test for Emulsified Asphalts – Particle Charge”

2.6.9.1. Other Equivalent or Closely Similar Standards

ASTM D7402

2.6.9.2. Applicable Materials and Test Conditions

- Cationic emulsified asphalt, polymer-modified cationic emulsified asphalt (Tables 8, 10)

2.6.9.3. Utility and Background of Test

This practice is used to verify whether an emulsion is cationic. Prior to testing, T59 requires the emulsions to be heated to 50±3°C and stirred thoroughly to ensure homogeneity. ASTM D7402 specifies an additional requirement for emulsions with viscosity testing requirements of 25°C to be heated and stirred at 25±3°C. The emulsion is poured in a 250-mL beaker up to a height that will allow the electrodes to be suspended approximately 25 mm in the emulsion. The electrodes are connected to a DC source, and the current is adjusted to at least 8 mA with a variable resistor. After 30 min from the start of the test or when the current drops to 2 mA, the current source is disconnected, and electrodes are washed with distilled water. The electrodes are observed for a discernible amount of asphalt deposit on them, and any evidence of a clearly discernible deposit on the cathode compared with the anode indicates that the emulsion will pass the test.

2.6.9.4. Advantages and Limitations of Test

Advantages:

- This test is simple, easy to conduct, fast, and requires low capital cost in terms of equipment.
- The results produced by this test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- As stated in T59 standard, this test method requires subjective evaluation of the test results, and no precision and bias statements are specified for this test method thus far.

2.6.10. Tex-542-C “Determining Breaking Index for Asphalt Emulsions”

2.6.10.1. Other Equivalent or Closely Similar Standards

None.

2.6.10.2. Applicable Materials and Test Conditions

- PM emulsified asphalt, PM cationic emulsified asphalt (Tables 9, 10)

2.6.10.3. Utility and Background of Test

This test is an indication of the stability of RS emulsified asphalts. The amount of a siliceous filler to break 100 g of the emulsion is measured and reported as the breaking index.

2.6.10.4. Advantages and Limitations of Test

Advantages:

- This test is simple, easy to conduct, fast, and require low capital cost in terms of equipment.
- The results produced by this test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- No correlation has been found between the breaking index values obtained from laboratory tests and the actual breaking process in the field.

2.7. Cutback Tests

2.7.1. D95 “Water Content”

2.7.1.1. Other Equivalent or Closely Similar Standards

American Petroleum Institute’s Manual of Petroleum Measurement Standards, Chapter 10.5

2.7.1.2. Applicable Materials and Test Conditions

Applicable Materials:

- Rapid-Curing (RC), Medium Curing (MC) and Special-Use (SU) Cutback Asphalts (Tables 4, 5, and 6 of Item 300)

Test Conditions:

- Use aromatic liquids (such as petroleum or coal tar Naphtha; industrial grade Xylene; 20% industrial grade Toluene + 80% industrial grade Xylene) that can dissolve asphalt, bitumen, tar, and related products (Table 1 of D95).

2.7.1.3. Utility and Background of Test

The test material and a water-immiscible (but asphalt-miscible) solvent is heated together in a glass or a metal still. The evaporated solvent and water are condensed back to liquid state by a reflux condenser and collected in a glass trap. The volume of water is read from the graduated section of the trap and used to calculate %water.

This test is typically used with cutbacks to ensure that there is no water contamination. Water would create foaming when you heat a cutback for application. Foaming can be a safety hazard if the cutback is being heated for application. This test is rarely performed from a quality assurance perspective. In fact, if a distillation test is conducted on a water contaminated cutback, the material will also foam during the test due to the presence of water.

2.7.1.4. Advantages and Limitations of Test

General:

Identifies excess water content that might cause a foaming-related safety hazard.

Advantages:

- Sample preparation (preparing a solution) and testing steps (heat, condense and collect) are straightforward and easy to follow and execute.
- The parameter obtained from the test is easy to measure (the volumes are read directly from graduated trap) and interpret (higher %water ➔ chance of foaming).
- The capital cost of the equipment is not very high (heater + still + condenser + receiver = approx. $1500).

Limitations:

- The test method does not specify the exact amount test material to be dissolved in the solvent. It only specifies the exact amount of solvent.
- Safety Issues:
    - Industrial grade Xylene is flammable and has harmful vapor.
    - Petroleum or coal tar Naphtha is extremely flammable and has a harmful vapor.
- Chances of errors in %water calculation:
    - The volatile components of the test material might be soluble in water and might artificially increase its value.

- The water droplets that adhere to the surfaces of the condenser, the receiver, etc., might artificially decreasing its value.
- The water content of the solvent can artificially increase its value. [To avoid it, determine the water content of the solvent using the exact method without asphalt in it and subtract its contribution to total water content.]

2.7.2. T78 "Distillation of Cutback Asphalt Products"

2.7.2.1. Other Equivalent or Closely Similar Standards

ASTM D402

2.7.2.2. Applicable Materials and Test Conditions

- Rapid curing, medium curing, and special use cutback asphalts (Tables 4, 5, 6)
- Specialty emulsions (Table 11)

2.7.2.3. Utility and Background of Test

This method is used to measure the amount of volatile constituents in cutback asphalts. 200 mL of the cutback is distilled at a controlled rate to a temperature of 680°F, and the volume of the distillate is measured at different temperatures to calculate the distillate fraction. The residue content in volume percent is also calculated as the difference between the original sample volume and the total distillate recovered up to 680°F. The test is also used to secure a sample of residual binder for testing.

2.7.2.4. Advantages and Limitations of Test

Advantages:

- The results produced by the test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- It is stated in AASHTO T78 that the presence of silicon in the cutback may affect the distillation residue by retarding the loss of volatiles after the residue has been poured into the residue container.
- Performing this test (including counting drops, temperature corrections, and recording volume of the distillate at various stages), as well as the sample preparation and the cleanup procedures, consume a significant amount of an operator's time and attention.

- It is likely that the formation of skin on the surface of the residue entraps vapors, and therefore causes higher penetration results. In such cases, T78 recommends to gently push aside the skin; however, this procedure may cause considerable disturbance to the sample.
- The test is a safety concern as the solvent (distillate) is naphtha/gasoline or kerosene boiling range solvent.

2.8. PG Tests

2.8.1. T313 "Determining the Flexural Creep Stiffness of Asphalt Binder Using BBR"

2.8.1.1. Other Equivalent or Closely Similar Standards

ASTM D6648

2.8.1.2. Applicable Materials and Test Conditions

- Polymer-modified AC (Table 3)
- PG binders (Table 17)

2.8.1.3. Utility and Background of Test

According to the AASHTO T313 standard, this test method determines the flexural creep stiffness or compliance of RTFO and PAV-aged asphalt binders by means of a bending beam rheometer. It can be used with unaged binders or with binders aged in accordance with AASHTO T240 or AASHTO R28, or both. The testing temperature ranges from -36°C to 0°C.

Some other pertinent information from the literature is summarized below:

- Recent studies have shown that the 4-mm parallel plate geometry for DSR can be used to obtain the BBR equivalent stiffness and m-value. The use of a DSR has been demonstrated as a useful method to characterize the low temperature behavior of asphalt binders due to improved efficiency and use of a significantly smaller amount of asphalt binder sample (25 mg) compared to the BBR method (15 g).
- The 4-mm parallel plate geometry is found to be reliable, fast, and simple to obtain the low-temperature rheological properties of the asphalt binder compared to the BBR method [55]. However, one major issue associated with this test method is that at low testing temperatures (below 5°C), the machine compliance can cause errors in

determining the correct modulus of asphalt binders [56]. This problem was resolved by including a correction for machine compliance, allowing testing asphalt binders to -40°C [57].

- Another issue with the 4-mm parallel plate method is the conversions that need to be made on the shear modulus to be comparable to BBR flexural measurements. This concern has been addressed by proposing some empirical functions that are based on the linear viscoelastic theory. Hajj et al. [58] showed that the DSR method can be used as surrogate for the BBR, provided that some caution be used in assuming the Poisson's ratio.

- The research team has also collected data to demonstrate that the BBR based stiffness and m-value can be replaced directly with the G* and delta parameter from the DSR test. The use of the DSR based parameters in lieu of converting these parameters to the BBR equivalent stiffness and m-value can significantly reduce time and errors in interpreting the test results.

2.8.1.4. Advantages and Limitations of Test

Advantages:

- The test duration is short (2 min from the start of loading time).

- Although the test requires some interpretation of the creep data to determine the stiffness and slope of the creep curve at 60-seconds, these analyses are typically automated and included in the software provided by most commercial manufacturers of BBR.

Limitations:

- This test method requires a relatively large amount of asphalt binder which may sometimes be difficult to obtain especially in the case of recycled and reclaimed materials. The sample size is also a constraint when considering the use of samples recovered from emulsions using methods, such as the moisture analyzer balance.

- The specimen pre-molding, the conditioning time in the bath, and the cleaning procedure occupy a significant amount of an operator's time and attention.

2.8.2. T314 "Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT)"

2.8.2.1. Applicable Materials and Test Conditions

- PG binders (Table 17)

2.8.2.2. Utility and Background of Test

In this test method, a direct tension device is used to apply a constant rate of elongation (1 mm/min) to the asphalt specimen and measure the failure stress and failure strain. When the binder specimen fails by fracture, the strain at failure is identified as the strain at peak load. When it fails without breaking, the strain at failure is the one corresponding to the maximum stress. If the failure strain is greater than 10%, the binder meets the requirements of AASHTO M320 for determining the critical cracking temperature.

In Item 300 (as well as in the original Superpave PG specifications), this test is recommended in certain situations where the binder has failed the stiffness criteria, but passed the m-value requirement from the BBR test, and the creep stiffness is between 300 and 600 MPa. In such situations, the direct tension failure strain requirement can be used instead of the creep stiffness requirement, as long as the m-value requirement is satisfied. The BBR based stiffness requirement is intended to ensure that when the service/environment temperature drops, the tensile stresses remain below a certain limit and do not exceed the tensile strength of the binder. However, if this requirement is not met, then it would be necessary to use the direct tension test to demonstrate that the tensile strength of the binder is higher than typical values, and therefore capable of withstanding the higher tensile stresses.

Some other pertinent information from the literature is summarized below:

- The direct tension test was originally developed and included in the specifications to address fracture properties for asphalt binders [59]. However, due to the numerous complications associated with the repeatability of the test, the most recent versions of PG specifications have made this test optional [60].

- Over the past decade, possible alternatives to this test have been proposed, such as the single-edge notch bending (SENB) test to better capture the damage resistance behavior of binders at low temperature. For instance, Tabatabaee et al. [61] found that the deformation at maximum load and fracture energy measurements from the SENB test can be good indicators for the low temperature behavior.

2.8.2.3. Limitations of Test

- High variability: This test requires a total of six specimens to be tested. The two lowest values of failures strain and stress are discarded, and the mean and standard deviation values from the remaining four specimens are calculated and reported. The need for this procedure arises because according to AASHTO T314, these two parameters are inherently variable for asphalt binders.

- Performing this test, as well as the sample preparation and the cleanup procedure, occupy a significant amount of an operator's time and attention.

2.8.3. T315 "Determining the Rheological Properties of Asphalt Binder Using DSR-Fatigue parameter"

2.8.3.1. Other Equivalent or Closely Similar Standards

ASTM D7175

2.8.3.2. Applicable Materials and Test Conditions

- PG binders (Table 17)

2.8.3.3. Utility and Background of Test

According to AASHTO T315, this test method is used to determine the complex shear modulus and the phase angle of RTFO and PAV-aged asphalt binders using the parallel plate test geometry of the DSR in dynamic (oscillatory) mode. These two rheological properties are used to determine the performance grade of asphalt binders in accordance with AASHTO M320. The fatigue parameter, $G^*\sin\delta$, is associated with intermediate temperature stiffness, and the intermediate temperature grade of an asphalt binder is determined based on the highest temperature that the fatigue parameter is less than 5000 kPa.

Some other pertinent information from the literature is summarized below:

- Because the Superpave fatigue parameter is only measured within the linear viscoelastic range of asphalt binders and after a much smaller number of load cycles than what pavement sections experience, many experts have argued that it cannot reliably predict the binder fatigue performance, and there is a substantial need for a test that can apply a sufficient amount of cycles to induce damage in order to obtain more realistic fatigue characterization [35]. In addition, the selection of this parameter was based on controlled strain conditions relevant for thin pavement layers.

- To overcome the limitations of the current specifications, NCHRP in 2001 [62] introduced the time sweep (TS) test, allowing for measuring the fatigue behavior of binders beyond the linear viscoelastic range (LVE). A serious drawback with this test is the time-consuming procedure associated with the repeated cyclic loading at a fixed amplitude. This limitation led experts to investigate a new procedure, stress sweep, that applies an incrementally increasing repeated cyclic loading to the binder specimen, thus accelerating the damage accumulation, and reducing the test duration [63].

- In 2010 [64], a newly developed test, linear amplitude sweep (LAS), coupled with the simplified viscoelastic continuum damage (S-VECD) theory, was proposed to estimate the fatigue life of asphalt binders at any strain amplitude of interest [65]. This test applies systematically increased loading amplitudes to the binders, thus inducing damage, and providing better prediction of the fatigue life. Over the last few years, this test has been

evaluated by many researchers, and the results are controversial. In some cases, the results seems to be reasonable, but in other cases, the results are counterintuitive [66], and a reliable fatigue parameter is still needed.

2.8.3.4. Advantages and Limitations of Test

Advantages:

- This parameter can fairly evaluate the effect of aging on the stiffness and loss modulus of asphalt binders.

Limitations:

- This test is conducted at a fixed frequency (10 rad/s), and a relatively small number of load cycles within the LVE range, which does not reflect the actual fatigue performance in the non-LVE range.

2.8.4. T315 "Determining the Rheological Properties of Asphalt Binder Using DSR- Rutting Parameter"

2.8.4.1. Other Equivalent or Closely Similar Standards

ASTM D7175

2.8.4.2. Applicable Materials and Test Conditions

- Polymer-modified AC (Table 3)
- PG binders (Table 17)

2.8.4.3. Utility and Background of Test

According to AASHTO T315, this test method is used to determine the complex shear modulus and the phase angle of asphalt binders using the parallel plate test geometry of the DSR in dynamic (oscillatory) mode. These two rheological properties are used to determine the performance grade of asphalt binders in accordance with AASHTO M320. The rutting parameter, $G^*/\sin\delta$, is associated with high temperature stiffness, and the high temperature grade of an asphalt binder is determined based on the lower of the two temperatures that meet the M320 criterion for unaged ($G^*/\sin\delta > 1.0$ kPa) and RTFO-aged ($G^*/\sin\delta$>2.2 kPa) binders.

Some other pertinent information from the literature is summarized below:

- An increasing number of studies have found that the permanent deformation index currently used in the Superpave specification ($G^*/\sin\delta$) is not sufficient to characterize the performance of modified binders, and also does not provide accurate ranking of such

binders due to its deficiency in capturing the nonlinear response of the binders [7], [62]. To address this issue, researchers attempted to develop a new binder test, multiple stress creep recovery (MSCR), that can predict the binder performance more accurately in both linear and nonlinear regions, regardless of the modification type.

- Several studies have been performed to investigate the correlation of the Superpave rutting parameter and the MSCR test with the rutting behavior of mixtures, and all of those have demonstrated better coefficients of correlation for the latter, specifically for polymer modified binders. For instance, Laukkanen et al. [67] studied the capability of different rheological binder rutting indicators in predicting asphalt mixture rutting behavior, and found a stronger correlation between the non-recoverable creep compliance parameter (J_{nr3200}) from the MSCR test and the b exponent parameter derived from the power law model obtained from wheel tracking test on the mixtures. Hajj et al. also reviewed various new methods as alternatives to the current Superpave specification and reported acceptable consistency between the MSCR test results on the binders and the Hamburg Wheel Tracking Device test results on the mixtures [68].

2.8.4.4. Advantages and Limitations of Test

Advantages:

- The $G^*/\sin\delta$ parameter works well with characterizing the rutting susceptibility of unmodified asphalt binders.

Limitations:

- Several studies have reported poor correlation between this parameter and the rutting performance of certain binders, particularly polymer-modified binders, as well as the accumulated permanent strain in their respective mixtures. [69]
- This test is conducted at a fixed frequency (10 rad/s) within the LVE range which does not reflect the actual rutting that usually occurred in the non-LVE range.

2.9. Polymer Modification Tests

2.9.1. Tex-533-C "Determining Polymer Additive Percentages in Polymer Modified Asphalt Cements"

2.9.1.1. Other Equivalent or Closely Similar Standards

AASHTO T302

2.9.1.2. Applicable Materials and Test Conditions

- PM-AC (Table 3)

- PM emulsified asphalt, PM cationic emulsified asphalt (Tables 9, 10)

2.9.1.3. Utility and Background of Test

This test method uses Fourier Transform Infrared Spectroscopy (FTIR) and Attenuated Total Reflectance (ATR) techniques to determine the concentrations of various polymers in asphalt binder including: Styrene-Butadiene-Rubber (SBR), Styrene-Butadiene-Styrene (SBS) rubber, Ethylene Acrylic Acid (EAA), and Ethylene Vinyl Acetate (EVA).

Some other pertinent information from the literature is summarized below:

- In a Federal Highway Administration (FHWA) project conducted by Diefenderfer [70], the authors compared the FTIR method with the elastic recovery method (AASHTO T301) using a ductilometer and found both methods suitable to verify the presence of polymer with relatively low variability among the results. His study also showed that the elastic recovery results were more repeatable than the FTIR results.

- Kosinska et al. used a high-performance gel permeation/size-exclusion chromatography (GPC/SEC) technique to determine the modifier contents in polymer-modified binders and confirmed the viability of this technique as an alternative to FTIR while offering lower relative standard deviation (RSD) for the results [71].

- Ratajczak and Wilmanski evaluated the accuracy of two FTIR measurement modes: (1) transmission, and (2) attenuated total reflectance (ATR) in determining the polymer content of lab produced SBS modified binders and found that the former resulted in lower mean errors [72].

- Hofko et al. [73] investigated the repeatability and sensitivity of FTIR ATR spectra analysis by taking eight different analysis methods into consideration and found the smallest coefficient of variation for a method based on original spectra using the absolute baseline. They also concluded that in the case of SBS modified binders, the interference between the sulfoxide and butadiene band due to proximity impacts the analysis of changes in the sulfoxide structures.

2.9.1.4. Advantages and Limitations of Test

Advantages:

- The FTIR spectroscopy is a well-known technique and is widely used for different purposes in the asphalt industry.

- While other FTIR methods may be more accurate, the ATR method is faster for production testing.

Limitations:

- Accurate estimates can only be obtained using the method of standard addition, which requires preparation and laboratory blending of the base binder with a similar polymer in different concentrations. This process can be extremely labor- and time-consuming, and sometimes, it is very difficult to get the base binder.

2.9.2. Tex-540-C Polymer Separation Test

2.9.2.1. Other Equivalent or Closely Similar Standards

- ASTM D7173-16 (Polymer separation)
- AASHTO T 53 (softening point) and ASTM D36 (softening point)

2.9.2.2. Applicable Materials and Test Conditions

- Polymer-Modified Asphalt Cement (48 hours)

2.9.2.3. Utility and Background of Test

Approximately 350 grams of asphalt binder in a lidded can is removed from the oven after heating for 48 hours at 325°F. First, the upper surface of the binder is disturbed with a spatula, and the skimming or clumping of polymer are inspected. Second, the spatula is inserted all the way to the bottom of the can, and its consistency is inspected. Third, the sample is cooled down, the bottom of the can is cut, and the specimens sampled from the top and the bottom of the can are tested for softening points using AASHTO T53. The difference in their values is used to judge if there was polymer separation.

Asphalt binders that show significant clumping, skimming, and other changes during visual inspection and have significant differences in softening points at the top and bottom of the can have a higher tendency for polymer separation and vice versa.

Some findings from the literature pertinent to the scope of work are summarized below:

- Bahia and Anderson [2] suggested that softening point is an empirical single-point measurement.
- Yin et al. [74] used the samples obtained from the cigar tubes and the softening point values obtained from the ring-and-ball tests to study the storage stability (phase separation) of asphalt binders modified with recycled plastics [mainly recycled polyethylene (RPE) materials] with or without reactive copolymers. They found that RPE binders with certain dosages of copolymers passed the separation tests, while those without these copolymers failed the separation tests.

- Kim and Lee [75] used the samples obtained from the cigar tubes and the G*/sinδ values obtained from the DSR tests to investigate the phase separation of crumb rubber modified asphalts (CRMA) obtained from 12 different locations in 11 different storage conditions. They showed that the percent difference in G*/sinδ (a measure of phase separation) was sensitive to rubber concentration.

2.9.2.4. Advantages and Limitations of Test

General:

The test is used to evaluate phase separation (storage instability) in modified asphalt binders, especially those modified with latex rubber, SBS copolymers, ground tire rubber, or other alternative modifiers.

Advantages:

- The test method is easy to follow in terms of sample preparation (heat a can of binder for 48 hours, and cut the bottom of the can after cooling) and testing (run softening point test on the specimens obtained from the top and the bottom of the can).
- The measured parameter is straightforward to interpret (higher difference in softening point implied higher chance of polymer separation).
- The capital cost of equipment required to run this test is very low.

Limitations:

- Visually inspecting both the skimming or clumping of polymer in the upper layer of binder after disturbing it with a spatula and the consistency of binder after inserting the spatula all the way to the bottom is subjective.
- Softening point is not a fundamental rheological property of asphalt binder.
- Heating the sample for 48 hours, testing with spatula, letting it cool down to be able to cut the can at the bottom, performing softening point test, and then cleaning test apparatus (despite being easy task) all demand a significant amount of an operator's time and attention.
- A lot of material sample is required to conduct this test.
- The first part of the test is subjective, while the second part of the test is quite sensitive to extraneous factors such as vibrations, rate of heating, and the leveling of the instrument.

2.10. Rubber Modification Tests

2.10.1. Tex-544-C Rubber Content for Rubber-Asphalt Crack Sealant

2.10.1.1. Applicable Materials and Test Conditions

- RA crack sealers (Table 15)

2.10.1.2. Utility and Background of Test

This test method covers the determination of the percentage of tire rubber in rubber-asphalt crack sealers. Cores (31.75-38.10 mm in diameter) are drilled from the sealant sample using a core drill device and heated to a temperature of 350°F in a metal beaker. 300 mL of trichloroethylene is added to the sample and allowed to stand at room temperature for at least 4 hr., or until a complete separation between the asphalt and the rubber is seen. Then the sample is poured onto a No. 200 sieve, washed with clean trichloroethylene, and allowed to sit in a well-ventilated area for at least 30 min. The sieve is then placed in a 140°F oven for 30 min, and cooled down at room temperature for 15-20 min. The rubber content is then calculated as the difference between initial and final weight of the sieve divided by weight of the sample.

Some findings from the literature pertinent to the scope of work are summarized below:

- Zanetti et al. developed two different procedures to determine the crumb rubber (CR) content of asphalt rubber (AR) binders: (1) a comparative analysis of the chemical composition focused on the content of cobalt and antimony, and (2) analysis of the combustion products of the AR binders. They concluded that although both procedures provided satisfactory levels of accuracy and precision, considerable effort is still needed to perform calibration tests and minimize the sources of measurement errors [76].

2.10.1.3. Advantages and Limitations of Test

Advantages:

- The test is simple, easy to conduct, and requires low capital cost in terms of equipment.
- The results produced by the test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

Limitations:

- Trichloroethylene in the presence of heat and moisture can form acids, which are extremely harmful to health if inhaled. This risk is in addition to the inherent risks imposed by TCE if inhaled. It would be better to use a relatively safer solvent to determine the rubber content.

2.10.2. D5329 "Standard Test Methods for Sealants and Fillers, for Joints and Cracks in Asphalt Pavements – Bond Test"

2.10.2.1. Other Equivalent or Closely Similar Standards

- Tex-525-C
- AASHTO TP85

2.10.2.2. Applicable Materials and Test Conditions

- AR crack sealers (Table 15)

2.10.2.3. Utility and Background of Test

This test method is used to evaluate the adhesive failure of crack sealants. Mortar blocks are fabricated, placed on treated brass or TFE-fluorocarbon spacer strips, and spaced at the required width to form an opening for pouring the crack sealant. After sufficient cooling and removal of the excess material, the specimens are placed in a cold cabinet at the required temperature (20°F) for not less than 4 hr. Then the spacer blocks are removed, and the specimens are placed in an extension machine with a uniform extension rate of 3.0±0.3 mm per hour. Immediately after the extension, the specimens are examined for obvious separations within the sealant and between the sealant and the blocks.

Item 300 (Table 15, footnote 4) specifies that no crack in the sealing materials or break in the bond between the sealer and the mortar blocks over ¼ in. deep for any specimen is allowed.

Some findings from the literature pertinent to the scope of work are summarized below:

- Hu et al. [75] argued that the ASTM D5329 procedure is not only time consuming and usually takes several days to complete, but also, the pass/fail criterion is based on visual observation and the correlation between the bond test and the performance in the field is either weak or non-existent. To address these issues, they proposed a new laboratory method using an overlay tester (OT), and tested a total of 13 sealants for their adhesive behavior under cyclic loading (5 s loading time + 5 s unloading time). The results of their study indicated that the OT method can effectively differentiate between poor and good adhesive sealants; however, no field validations were performed to evaluate the correlation. Moreover, it was found that due to the temperature sensitivity of the crack sealants, the repeatability of the test protocol needed to be improved.
- Al-Qadi et al. [78] developed three laboratory testing approaches to measure the adhesion of cack sealers: (A) measuring the free energy of the bond, (B) using the direct tension tester (DTT) machine, and (C) using tests in conjunction with fracture mechanics to

compute metrics that are indicative of the adhesive bond strength. In summary, the DTT method was found to be the most practical method for routine use.

- In an attempt to establish better correlations between the lab results and the field measurements, and due to the limited availability of the DTT device in many DOTs, Sawalha et al. [79] developed an improved version of the existing crack sealer adhesion tester (CSAT) proposed by Al-Qadi et al. [78]. They introduced a change in the fixture geometry and showed consistent results with sufficient repeatability.

2.10.2.4. Advantages and Limitations of Test

Advantages:

- The test method is simple with a reasonable capital cost.

Limitations:

- The sample preparation and test duration occupy a considerable amount of a technician's time and elapsed time.
- The pass/fail criterion for this test is determined through visual observation [77].
- There is no evidence that the results of this test pertain to field performance.

2.10.3. ASTM D5329 "Standard Test Methods for Sealants and Fillers for Joints and Cracks in Asphalt Pavements - Resilience Test"

2.10.3.1. Other Equivalent or Closely Similar Standards

This document covers the resilience test portion of ASTM D5329 applicable for AR Binders.

2.10.3.2. Applicable Materials and Test Conditions

- AR Binders (Table 16)

2.10.3.3. Utility and Background of Test

This test method measures the ability of a sealant to recover after a steel ball has been forced into the surface. The standard penetrometer used for the penetration test can be used for this test. The only exception is that the needle is replaced with a ball penetration tool. The test is performed at 77°C. Similar to the penetration test, the ball penetration tool is released from the contact surface for 5 min, and the penetration is recorded. In the next step, the ball is pressed down into the specimen for an additional 100 units (10mm) at a uniform rate in 10 s and held constant on the specimen surface for an additional 5 s. Then the ball is released, and the specimen is allowed to recover for 20 s. The final dial reading is recorded, and the procedure is repeated at two other

points equally spaced from the first point. The percent recovery is computed, and the resilience is reported as the average of the three recovery measurements.

Some findings from the literature pertinent to the scope of work are summarized below:

- In a study on laboratory evaluation of crumb rubber modified binders and mixtures, Estakhri et al. pointed out that this test is typically used for joint sealants, and the relevancy of this test for AR binders is uncertain. They suggested that the resilience or "recoverability" offered by the aggregate and CR can be measured by a recovery test immediately following a creep test [80].

- The ability of an AR binder to recover from loads can also be evaluated by means of some recently developed rheological methods, such as a repeated creep test or an MSCR test. The MSCR test has been found to be a suitable method to evaluate the rutting potential of rubber modified binders at high temperatures [81].

- At intermediate temperatures, the fatigue behavior of AR binders can be characterized by the failure energy, which is measured as the area under the stress-strain curve of the linear amplitude sweep (LAS) test. The LAS test has been shown to be a simple and reliable technique for ranking the fatigue performance of different binders [82].

2.10.3.4. Advantages and Limitations of Test

Advantages:

- The test is simple, easy to conduct, fast, and requires low capital cost in terms of equipment.

- The results produced by the test are directly used, and no additional data analysis or interpretation is required to be performed by the operator.

- The test is mainly performed at 25°C (77°F), which is close to the average pavement service temperature.

Limitations:

- Slight variations while performing this test can cause large differences in the results. Errors are usually due to: (i) sampling and sample preparation, (ii) variations in temperature, and (iii) condition of the apparatus (straightness of the ball penetration tool, cleanliness, etc.).

2.11. Aging Tests

2.11.1. T179 "Effect of Heat and Air on Asphalt Materials (Thin-Film Oven Test)"

2.11.1.1. Other Equivalent or Closely Similar Standards

- ASTM D1754

2.11.1.2. Applicable Materials and Test Conditions

- AC (Table 2)
- PM cationic emulsions, rejuvenating agents (Table 10)
- AR binders (Table 16)

2.11.1.3. Utility and Background of Test

The thin film oven (TFO) test simulates the aging process which asphalt binder undergoes during the conventional hot mixing. The test is performed on thin asphalt films for 5 hr. at 163°C.

Some findings from the literature pertinent to the scope of work are summarized below:

- The TFOT test was first introduced by Lewis and Welborn to simulate the aging of asphalt binders during plant mixing. Later, this test method was criticized by researchers due to major limitations, such as excessive film thickness and nonuniform aging [83]. To address these issues, several methods were proposed by researchers, including the RTFO test developed by the California Division of Highway that gained considerable attention and was incorporated into the Superpave PG specification to simulate short-term aging of asphalt binders [84].
- Shiau et al. [85] conducted a study to assess the effect of RTFOT and TFOT on 20 commonly used asphalt binders in Florida. The study included penetration, absolute viscosity, and infrared spectroscopy tests on the binders before and after aging, and comparisons were made at three temperature levels (285°F, 325°F, 365°F). Results from the penetration and the viscosity tests indicated that aging simulated by RTFOT was relatively more severe at 285°F and 325°F; however, no significant difference was observed at 365°F. Moreover, the carbonyl ratio values obtained using the infrared spectroscopy test indicated that there was no significant difference due to aging at 285°F and 325°F; while at 365°F, the TFOT appeared to result in slightly more severe aging. This apparent anomaly could be due to the fact that rheological indicators represent the combined effect of both loss of volatiles and oxidative aging, whereas carbonyl area from

FTIR spectroscopy reflects only the effect of oxidation. In the case of short-term aging, both loss of volatiles and oxidation contribute to aging, and therefore, it is likely that the results based on rheological indices are more accurate.

- In another experimental study conducted by Lu and Isacsson [86], the differences between these two aging methods were investigated in terms of the chemistry and the rheology of seven different bitumen, and the results indicated a strong correlation between the two methods.

2.11.1.4. Advantages and Limitations of Test

Advantages:

- Compared to the RTFO method, this method results in less waste of the residue (scraping is eliminated).
- In cases where the mass loss/gain is within the acceptable range (<0.4%), the TFOT pans can be directly placed in the PAV apparatus for further aging.
- The TFOT can also be used to age binders that have a very low viscosity at the aging temperature and are prone to flowing out of the bottle.

Limitations:

- The 5-hour testing period, compared to the 80 min RTFO test, is the major limitation of this test method.
- It has been reported that sometimes the binder being aged through this test method forms a skin over the sample, thus reducing the homogeneity and inhibiting the aging process.

2.11.2. T240 "Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test)" and Tex-541-C "Rolling Thin Film Oven Test for Asphalt Binders"

2.11.2.1. Other Equivalent or Closely Similar Standards

ASTM D2872

2.11.2.2. Applicable Materials and Test Conditions

2.11.2.2.1. T240

- PM cationic emulsified asphalt (Table 10)

2.11.2.2.2. Tex-541-C

- PM AC (Table 3)
- PM emulsified asphalt (Table 9)
- PG binders (Table 17)

2.11.2.3. Utility and Background of Test

The Tex-541-C procedure is similar but not identical to the T240 procedure. The main function of both procedures is to simulate aging of asphalt binder during plant mixing and to provide residue for additional testing. The slight variations between the two procedures are listed below:

- According to T240, a cooling period of a minimum of 60 min and a maximum of 180 min is needed after pouring the asphalt samples into the bottles and before loading them into the carriage. However, Tex-541-C requires the bottles to be loaded in the carriage as soon as possible after pouring.
- The air flow to the oven is set to 4±0.3 L/min in T240, while in Tex-541-C, it is set to 4±0.2 L/min.
- The test temperature in T240 is 163±1.0°C, while in Tex-541-C, it is 163±0.5°C.
- The test duration in T240 is strictly set to 85 min, while Tex-541-C allows a 5 min tolerance. This is because according to T240, the containers shall be removed from the oven one at a time while the remaining containers are still rotating in the carriage and the air and heat are continually being applied. Tex-541-C, however, leaves the carriage off after removing the first non-mass-change bottle from the oven.
- If the mass change is being determined, T240 requires a cooling period of at least 60 min and no more than 180 min for the designated bottles. Tex-541-C only states a safe handling temperature as a requirement.

Some findings from the literature pertinent to the scope of work are summarized below:

- Several publications have appeared in recent years documenting the shortcomings of the RTFO method to simulate short-term aging for polymer-modified asphalt (PMA). Many of these studies suggested that the aging temperature should be increased to an extent that the high viscosity PMA binders have a low viscosity that will allow them to roll and age uniformly in the bottles [87].
- Other studies proposed new test methods such as Modified German Rotating Flask (MGRF) [88], Stirred Air Flow Test (SAFT) [89], and Rotating Cylinder Aging Test [90]. Later, NCHRP [91] investigated the viability of MGRF and SAFT as potential

replacements of the RTFO and concluded that MGRF could be considered as an acceptable replacement.

- A number of studies have also been conducted to propose modifications to the existing RTFO test method. For instance, Bahia [92] suggested two modifications: (1) using a number of steel spheres, and (2) using a steel rod to force the spreading of binder. Although both methods appeared to be viable alternatives to the conventional RTFO method, later studies indicated that the problem with uniform aging was not completely resolved [93].

2.11.2.4. Advantages and Limitations of Test

Advantages:

- The RTFO test is still the most commonly used standard test to simulate the short-term aging of binders. Compared to the thin film oven test (TFOT), it is faster and more efficient procedure.
- Other procedures, such as MGRF, may improve accuracy and uniformity of aging, but do not really provide any substantial advantage in terms of ease of aging, time required to age, or preparatory work before and after aging. Additionally, the MGRF can only age one binder at a time, while the RTFO can hold eight bottles and consequently age several different binders simultaneously.

Limitations:

- In the case of high-viscosity binders such as polymer modified binders, this test has appeared to provide insufficient and non-uniform aging.

2.11.3. R28 "Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)"

2.11.3.1. Other Equivalent or Closely Similar Standards

ASTM D6521

2.11.3.2. Applicable Materials and Test Conditions

- PG binders (Table 17)

2.11.3.3. Utility and Background of Test

This test method is used to simulate the in-service oxidative aging of asphalt binders by means of pressurized air and elevated temperatures, and is intended for use with residue from T240 (RTFO) test.

A specified amount of RTFO residue (50 g) is placed in stainless steel pans and aged at 100°C for 20 hr. at an air pressure of 2.10 MPa. At the completion of this test, the residue is vacuum degassed and ready for further testing.

Some findings from the literature pertinent to the scope of work are summarized below:

- In the SHRP-A-369 report, Anderson et al. performed a field validation study to verify that the changes in the physical and chemical properties of PAV-aged binders are similar to the changes that occur during long-term aging in the field. Their study included a large number of sections varying in location and type of asphalt used. They concluded that temperature was the most influential factor that controls the rate of aging in both scenarios; therefore, the PAV testing temperature should be selected in a manner to reflect the climate conditions [94].

- In 2005, Glover et al. [36] proposed a new aging procedure using the PAV with thinner film thickness (0.86 mm) and longer aging time (32 hr.). They demonstrated that any change in the length of the test, as well as the temperature and pressure, will change the relative rankings of asphalt binders; therefore, replacing long-term aging in the field with a laboratory test at higher temperatures and pressures may result in false rankings. To address this issue, they simulated the long-term aging in an environmental room held at 60°C (close to Texas road conditions during summer) and 1 atm air, and compared all other aging conditions using PAV as to relative ranking with results from the environmental room. The results showed that 32 hr. at 90°C and 20 atm air in PAV corresponded well to 122 days aging in the environmental room. The current PAV protocol also agreed well with 38 days of aging in the environmental room.

- Glover et al. in their literature review on evaluation of binder aging and its influence on aging of HMA pointed out that the 20 hr. period of PAV oxidative aging may not necessarily correspond to in-service aging time due to the effect of other factors, such as climate, binder kinetics, and mixture properties, that are not considered during laboratory long-term aging. Therefore, they recommended development and calibration of a laboratory test that ages binders to a level that can better represent the long-term aging in the field [95].

- Kaveh and Hesp used infrared techniques to study the effect of variations in the PAV test temperatures, aging time, film thickness, and the presence of moisture on the relative degree of chemical aging in seven asphalt cements. They compared the carbonyl,

sulfoxide, and butadiene indices for the RTFO+PAV aged binders with those for the binders which were recovered after eight years of service, and found that using a reduced film thickness or extended aging time could better resemble changes due to aging in the field [96].

2.11.3.4. Advantages and Limitations of Test

Advantages:

- This test is simple, efficient, and easy to conduct.

Limitations:

- As stated in AASHTO R28, the aging of asphalt binders during service is affected not only by the temperature and the climatic conditions, but also by various mixture-associated variables, such as aggregate properties and permeability of the mix, which are not accounted in this method.
- Previous research on the PAV film thickness has shown that, due to diffusion resistance, samples may not achieve uniform aging, and thus may not accurately represent the long-term aging in the field [36][97].

2.12. Summary

The background on the tests included in Item 300 specifications has been presented in this chapter. Based on the review and discussions/workshops with the industry, changes to the Item 300 were recommended. Researchers categorized the recommended changes in the following three levels for adoption:

- Level 1: Proposed changes that have adequate evidence based on the review of the literature, data analysis, and stakeholder input shall be recommended for immediate adoption.
- Level 2: Proposed changes that have substantial evidence based on the findings from the previous task, but that require revised parameters or specification limits shall fall into this category. For example, a different but well-established or recognized method may be used to measure or replace viscosity measurement for a specific product in Item 300. This shall entail change in the specification limits provided in Item 300 to match the proposed method. For such scenarios, a testing plan shall be developed with materials from diverse sources of the specific product to generate data that can inform or validate the specification limits based on the proposed parameter.
- Level 3: Proposed changes that require additional data/evidence for adoption, as well as validated specification limits, shall fall into this category. For the proposed and

prioritized changes in this category as identified in Task 2 with the Receiving Agency, the researchers shall develop a detailed testing plan to collect data that can be used to develop informed specification limits.

A summary of the recommended changes along with the adoption level is shown in Table 2.2.

Table 2.2. Recommended Test Method Changes.

	Test method	Standard	Category	Readiness Level
Stiffness	Penetration	AASHTO T49	Remove/ Replace	3
	Softening Point	AASHTO T53	Replace	3
	Ductility and Elastic Recovery	AASHTO T51 @ 39.2°F	Remove	1
		AASHTO T51 @ 77°F	Replace	3
Viscosity	Kinematic Viscosity	AASHTO T201	Replace	2
	Apparent Viscosity	AASHTO T202	Replace	2
	Saybolt Viscosity	AASHTO T72	Replace	1
	Apparent Rotational Viscosity	ASTM D2196	Replace	3
PG Tests	BBR (stiffness, m-value, ΔTc)	AASHTO T313	Retain/ Renovate	2
	Direct Tension	AASHTO T314	Remove	1
	DSR fatigue parameter	AASHTO T315	Renovate	3
Polymer Modification	Polymer Separation	Tex-540-C	Replace	2
Aging	Thin Film Oven Test	AASHTO T179	Remove	1
Emulsion Tests	Emulsion Recovery	AASHTO T59	Replace	3
Cutback Tests	Cutback Recovery	AASHTO T78	Replace	3

The next chapter summarizes the drafts of the new test methods developed (adoption level 2 and 3) during this project, and Chapter 4 discusses and analyzes the data collected using these test procedures.

Chapter 3. New Test Procedures and Proposed Changes for Item 300

This chapter summarizes the draft of the new test procedures developed during this project.

3.1. Tex-540-C Polymer Separation in Modified Asphalt Systems

3.1.1. Goal

The main goal of this exercise was to replace Tex-540-C with ASTM D7173 or a similar method in combination with a DSR based parameter in lieu of the softening point parameter. This test is currently being performed on polymer modified AC binders (Table 3) and PG binders with UTI greater than 86 (Table 17). According to Table 3 of Item 300, the allowable separation is "None" when testing using Tex-540-C. In practice, the pass/fail limit for the percentage difference in softening point (per Tex-540-C) between a sample obtained from the top and bottom of a can after conditioning is set as 5%. Samples showing more than this difference are considered to have failed the separation test. However, when shifting to a DSR based parameter, there is a need to identify the appropriate parameter as well as the acceptable difference based on the acceptable variability of the parameter.

3.1.2. Parameters

After evaluating various DSR based parameters, $G^*/\sin\delta$ was identified to be the most appropriate parameter to quantify the separation.

3.1.3. New test method

The draft version of the new test procedure can be found in Appendix A.

3.2. AASHTO T59 and T78 Emulsion and Cutback Recovery

3.2.1. Goal

The main goal of this exercise was to research, develop, and establish residue recovery methods that can be used with cutbacks and emulsions. This method is intended to provide a sample of the binder for further performance testing. It is not intended to provide a compositional analysis, as is the case with other typical methods used with cutbacks.

3.2.2. Parameters

Testing of the residue obtained from the procedure described in the next section was performed using a DSR using the following protocol:

- Test emulsion residues using a DSR and 25 mm parallel plate (perform all tests below on the same sample in the order described below):
    - G*/sin δ at 64°C (300 s equilibration time) at 10% strain and 10 rad/s for ten cycles
    - MSCR at 64°C (only for polymer modified emulsions)
    - G*/sin δ at 25°C (300 s soak time and 600 s equilibration time) at 1% strain and 0.1 rad/s for ten cycles
- Test cutback residues using a DSR and 25 mm parallel plate using the following protocol (perform all tests below on the same sample in the order described below):
    - G*/sin δ at 64°C (300 s equilibration time) at 10% strain and 10 rad/s for ten cycles
    - G*/sin δ at 25°C (300 s soak time and 600 s equilibration time) at 1% strain and 0.1 rad/s for ten cycles

The average value of G*/sin δ parameter obtained from the last five cycles at 64°C and 25°C was reported. For polymer modified emulsions, MSCR parameters along with the G*/sin δ parameter was reported.

3.2.3. New test method

The draft version of the new test procedure can be found in Appendix B.

3.3. AASHTO T201 Kinematic Viscosity of Asphalts and AASHTO T72 Saybolt Viscosity

3.3.1. Goal

The main goals of this exercise were to:

- Replace T201 with rotational viscometer. This test is currently being performed at 140°F on RC, MC, and special-use cutbacks (Tables 4, 5, 6), as well as rejuvenating agents (Table 10).

- Replace T72 with rotational viscometer. This test is currently being performed at 77°F or 122°F on all type of emulsions (Tables 7 to 11), and at 77°F on recycling agents and emulsified recycling agents.

3.3.2. Parameters

Average viscosity obtained at three one-minute intervals by using the procedure as described in the developed test procedure was reported.

3.3.3. New test method

The draft version of the new test procedure can be found in Appendix C.

3.4. 8mm DSR for low temperature testing of PG binders

3.4.1. Goal

The main goal of this exercise was to track the use of G* and δ at low temperatures measured using an 8 mm DSR as a potential alternative to BBR S and m values.

3.4.2. Parameters

Average of G* and δ from the last five cycles at the PG low temperature grade plus 10°C was reported.

3.4.3. New test method

The draft version of the new test procedure can be found in Appendix D.

3.5. Poker-chip test for intermediate temperature testing of PG binders

3.5.1. Goal

The main goal of this exercise was to track the poker chip strength and ductility parameter at intermediate temperature in order to use them as a potential alternative to G*sin(δ) fatigue parameter at intermediate temperature.

3.5.2. Parameters

Poker chip strength and ductility values obtained from the developed test procedure were reported.

3.5.3. New test method

The draft version of the new test procedure can be found in Appendix E.

Chapter 4. Development of the New Test procedures in Item 300

This chapter presents and analyses the data collected for the new test procedures developed in this project.

4.1. Tex-540-C Polymer Separation in Modified Asphalt Systems

4.1.1. Goal

The goal for developing this new test procedure, was stated in 3.1.1. This section describes the work performed to develop/modify a test procedure and method of determining unacceptable separation of polymer.

4.1.2. Scope

The method is intended to be applied to the materials listed below. The numbers included in the third column indicate the approximate testing load based on a 12-month analysis of the LIMS data).

Table 4.1. Polymer separation testing load based on LIMS data.

TABLE	ITEM	TESTS/YR**
Table 3: PM AC	AC-15P	54
	AC-20XP	43
	AC-10-2TR	8
	AC-20-5TR	64
	AC-12-5TR*	6
Table 17: PG binders	64-22	1
	64-28	54
	64-34	4
	70-22	198
	70-28	75
	76-22	193
	76-28	62
	82-22	10

* AC-5+SBR and AC-10+SBR are not shown because these were slated to be removed; AC-12-5TR is shown because it was added in Table 3 of Item 300.

** Based on 2020 LIMS data.

4.1.3. Evaluation

4.1.3.1. Method

The separation test was carried out using the cigar tube test according to ASTM D7173. The main steps in the test method are summarized below:

1. "Cigar tubes" are basically aluminum shell casings for packaging applications. The tubes are 5.5 inches in height and 1 inch in diameter and are available commercially[1]. A tube rack is used to hold the tubes in vertical position (Figure 4.1) during conditioning[2]. Figure 4.1 shows tubes and a tube rack.
2. The binder sample is heated in the oven at 163 ± 5°C until it is sufficiently fluid to pour. The sample is then stirred thoroughly in the container, and 50±0.5 g of the sample is poured into each tube (Figure 4.2).
3. The tubes are then sealed by folding the excess tube over two times and crimping tightly (Figure 4.3).
4. The sealed tubes are then immediately placed in the oven at 163 ± 5°C for a period of 48 ± 1 hour. At the end of the conditioning period, the tubes are removed from the oven and immediately placed in a freezer at -10 ± 10°C for a minimum of 4 hours.
5. The tubes are then removed and cut into three portions of approximately equal length. The center portion is discarded, and the top and bottom portions are placed in separate covered containers (Figure 4.44.).
6. The containers are placed in the oven at 163 ± 5°C until the sample is sufficiently fluid to remove the pieces of aluminum tube but no longer than 30 minutes.
7. The aluminum pieces are removed, and the samples are stirred thoroughly before being used for DSR or other testing (Figure 4.5).

[1] One possible source for tubes that was used in this study:
https://www.hmalabsupply.com/products/5-50-x-1-diameter-aluminum-tube-box-of-144-tubes

[2] One possible source for tube rack that was used in this study:
https://www.amazon.com/dp/B00T57R09S/ref=sspa_dk_detail_1?psc=1&pd_rd_i=B00T57R09Sp13NParams&spLa=ZW5jcnlwdGVkUXVhbGlmaWVyPUE3NThaT1daTVY5M0MmZW5jcnlwdGVkSWQ9QTA5NTIzMTIxTU9BUTUwTTdHMlM0JmVuY3J5cHRlZEFkSWQ9QTAyNzcyNjZIN0FHTlVFNVpBMjMmd2lkZ2V0TmFtZT1zcF9kZXRhaWwyJmFjdGlvbj1jbGlja1JlZGlyZWN0JmRvTm90TG9nQ2xpY2s9dHJ1ZQ==#customerReviews

Figure 4.1. "Cigar Tubes" in a stand.

Figure 4.2. Sample being poured into the cigar tube.

Figure 4.3. Cigar tube with sample; top of the tube is folded and crimped by hand to seal the sample.

Figure 4.4. Cigar tube is cut into three after removing from the freezer (left: before cutting, middle: after being cut into three parts, right: top and bottom parts placed in cans).

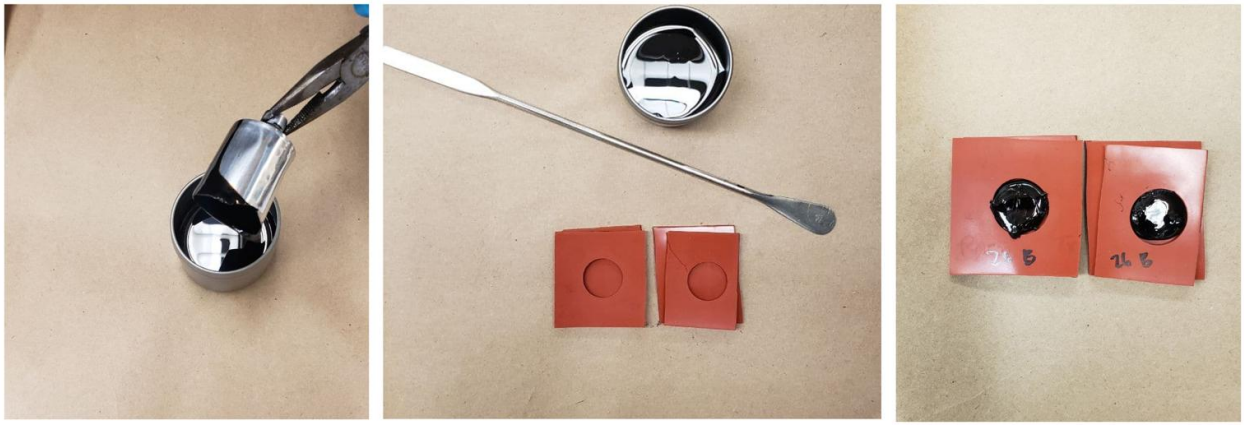


Figure 4.5. Left: aluminum tube is removed from heated can; middle and right: the residual binder is transferred to a mold for further testing.

4.1.3.2. Materials and Tests

A range of polymer modified AC and PG binders were selected for this study. The asphalt producers were also requested to provide samples that were intentionally designed to potentially exhibit separation. A list of these binders is provided below.

- PG 70-28 x 1 different source x 2 different propensities to separate (Sample IDs: PS05, 06)
- AC-15P x 1 source x 1 condition (Sample ID: PS07)
- AC-20-5TR x 2 different sources x 2 different propensities to separate (Sample IDs: PS09, 10, 11, 12)
- Two random samples from the QM stream that failed Tex-540-C (Sample IDs: A, B).

The materials were contributed by producers who typically supply materials for projects within the state of Texas. Each binder was split into separate quart cans for each participating

laboratory. In the case of the two random samples that were obtained from the QM stream and failed separation test (Tex-540-C), the sample obtained was from the leftover material from QM testing. This sample was heated, thoroughly stirred, and split into two parts, one for each participating laboratory.

Table 4.1 and Table 4.2 present the laboratories that participated and the parameters that were collected by each laboratory using the two different separation methods (Tex-540-C and ASTM D7173) and subsequent tests for separation (softening point and DSR based parameters). The tests were performed after allowing the separation for 5 hours and 48 hours, respectively. Only results from the 48-hour separation tests are presented here.

Table 4.1. Testing program for separation test showing number of replicates tested at different locations for Tex-540-C.

Agency →	TxDOT MTD	Lab 1 (Producer)	Lab 2 (Producer)	Lab 3 (Research)	Lab 4 (Research)
Test Performed →	Tex-540-C + Softening Point				
PG 76-22 PS01	2	2	2		
PG 76-22 PS02	2	2	2		
PG 76-22 PS03	2				
PG 76-22 PS04	2				
PG 70-28 PS05	2				
PG 70-28 PS06	2				
AC 15-P PS07	2				
AC-20-5TR PS09	2	2	2		
AC-20-5TR PS10	2	2	2		
AC-20-5TR PS11	2	2	2		
AC-20-5TR PS12	2	2	2		
PG 76-22 A	2^{+}				
PG 76-22 B	2^{+}				

Table 4.2. Testing program for separation test showing number of replicates tested at different locations for ASTM D7173.

Agency →	TxDOT MTD	Lab 1 (Producer)	Lab 2 (Producer)	Lab 3 (Research)	Lab 4 (Research)
Test Performed →	ASTM D7173 + G*/sin δ; Jnr and Elastic Recovery from MSCR test at 0.1 and 3.2 kPa*				
PG 76-22 PS01		2	2	2	2
PG 76-22 PS02		2	2	2	2
PG 76-22 PS03			1	2	2
PG 76-22 PS04			1	2	2
PG 70-28 PS05			1	2	2
PG 70-28 PS06			1	2	2
AC 15-P PS07			2	2	2
AC-20-5TR PS09		2	2	2	2
AC-20-5TR PS10		2	2	2	2
AC-20-5TR PS11		2	2	2	2
AC-20-5TR PS12		2	2	2	2
PG 76-22 A				2	2
PG 76-22 B				2	2

* Tests were performed at 64C for AC binders and high temperature grade for PG binders

4.1.4. Results and Discussion

In all cases, the results are expressed as the percentage difference (from the average) in the measured value between the top and bottom tubes calculated as shown below:

$$\%\ \text{Difference} = \frac{|Top\ -\ Bottom|}{(Top\ +\ Bottom)/2}$$

Figure 4.6 compares the results (% difference) from the current Tex-540-C and softening point from MTD versus independent measurements from two different producers. This figure shows that for this set of binders, the results were mostly consistent across the different laboratories.

For the remainder of this discussion, results from Tex-540-C measured at MTD will be used as the basis.

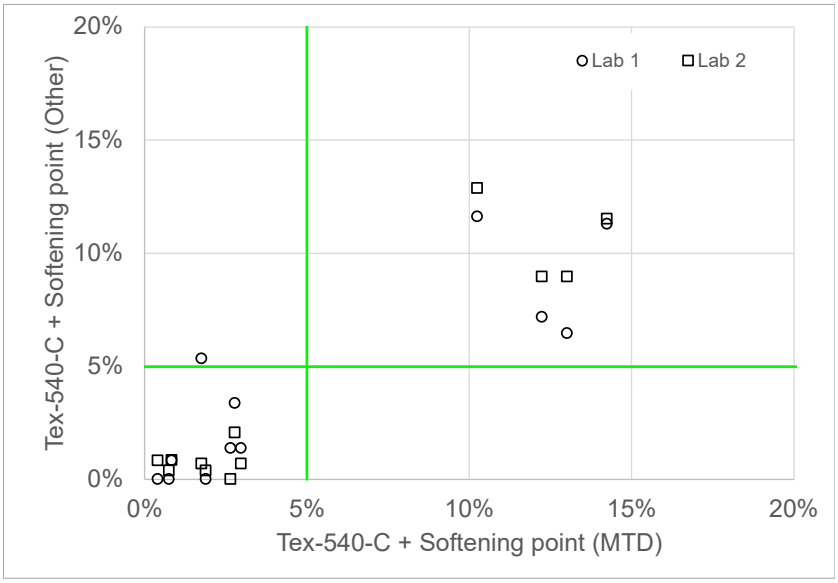


Figure 4.6. Comparison of Tex-540-C combined with softening point results (% difference) from MTD and two producer labs.

Figure 4.7 through Figure 4.11 compare the results (% difference) from ASTM D7173 and five different parameters (G*/sin δ, Elastic Recovery at 0.1 and 3.2 kPa, and Jnr from 0.1 and 3.2 kPa from the MSCR test) to the results measured using Tex-540-C at MTD. A cut-off at 5% is shown for the results from Tex-540-C based on the current practice, and a cut-off at 20% is shown for the other parameters being compared. The latter is tentative and only intended to facilitate the discussion. Replicate data was not averaged and is shown as-is in these figures. A few important observations are as follows:

- Data in the top-right and bottom-left quadrants are for binders that fail or pass in both scenarios consistently, respectively.
- Considering ASTM D7173 and G*/sin δ as the parameter, the results show that most binders pass or fail consistently across both parameters. A few data fall in the bottom right quadrant, i.e., binders that fail the Tex-540-C, but do not fail the proposed G*/sin δ (Figure 4.7).
- Considering ASTM D7173 and Elastic Recovery at 0.1 kPa from the MSCR test as the parameter, the results show that most binders pass or fail consistently across both parameters, although a few data fall on the 20% border for the MSCR parameter (Figure 4.8). A few data in the bottom right quadrant correspond to PS09. Interestingly, this binder showed low separation across all rheological indices and only showed some

difference in the softening point parameter. Further, about four points fall on the top-left quadrant, i.e., fail the elastic recovery requirement, but pass softening point. There was no systematic trend for these points (two points were from one lab, whereas three labs consistently showed that there was separation based on elastic recovery, and the other two points were isolated replicates). However, when this same comparison is made with Elastic Recovery at 3.2 kPa from the MSCR, there were several binders that failed the elastic recovery requirement, but passed the softening point, i.e. points in the top-left quadrant (Figure 4.9). A closer examination of the data revealed that these points correspond to the AC binders with very low elastic recovery (<10%). As such, even small variations in the measured elastic recovery resulted in large percentage difference.

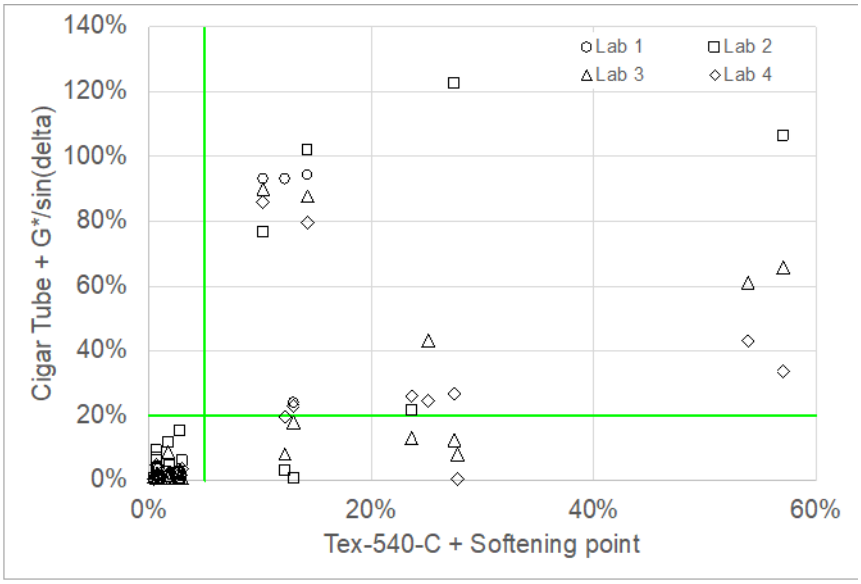


Figure 4.7. Comparison of Tex-540-C and Softening Point with ASTM D7173 and G/sin δ at high PG temperature or 64C for AC binders.*

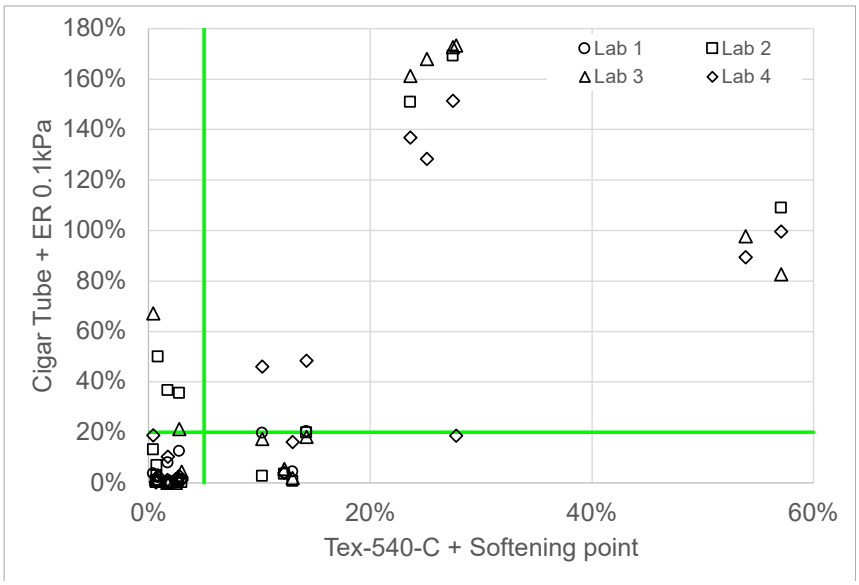


Figure 4.8. Comparison of Tex-540-C and Softening Point with ASTM D7173 and MSCR Elastic Recovery at 0.1 kPa at high PG temperature or 64C for AC binders.

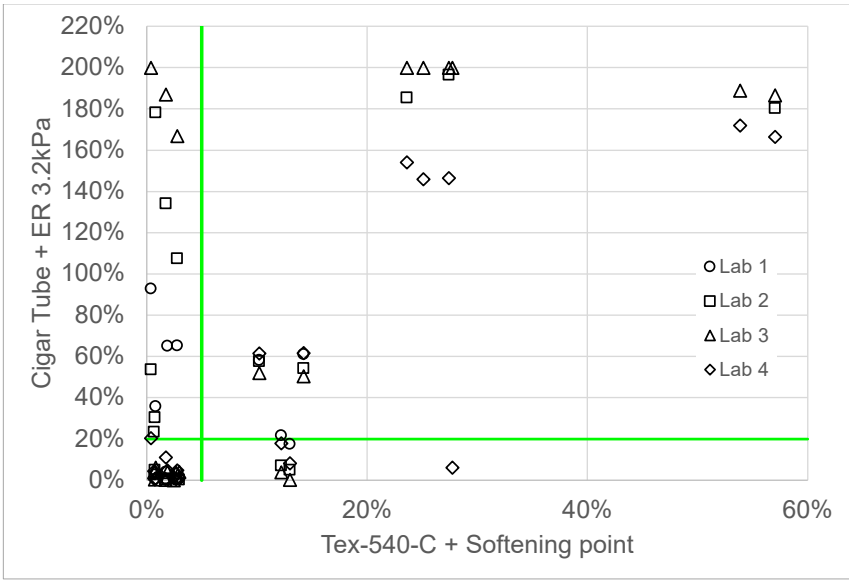


Figure 4.9. Comparison of Tex-540-C and Softening Point with ASTM D7173 and MSCR Elastic Recovery at 3.2 kPa at high PG temperature or 64C for AC binders.

Figure 4.10 and Figure 4.11 present comparisons of results from Tex-540-C with the ASTM D7173 and Jnr from the MSCR test at 0.1 and 3.2 kPa, respectively. The results from this parameter were similar to the results from the elastic recovery parameter.

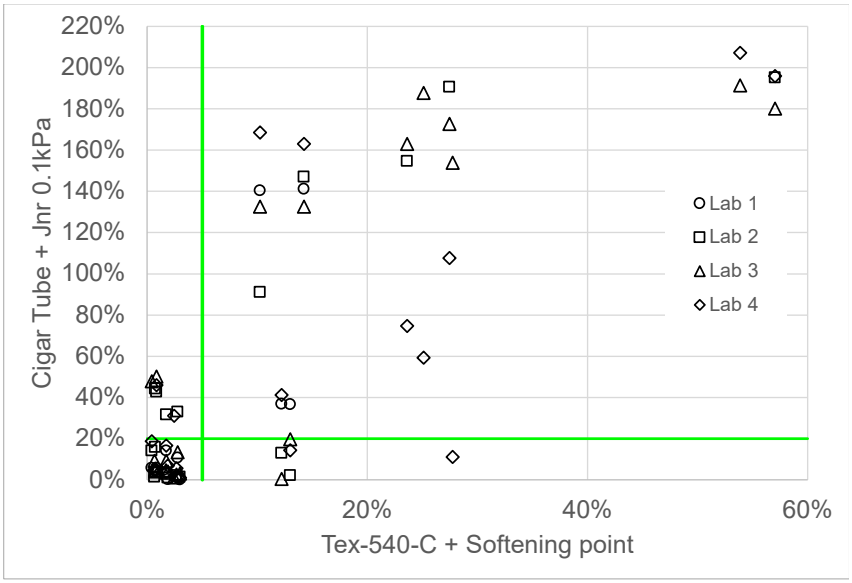


Figure 4.10. Comparison of Tex-540-C and Softening Point with ASTM D7173 and Jnr from MSCR test at 0.1 kPa at high PG temperature or 64C for AC binders.

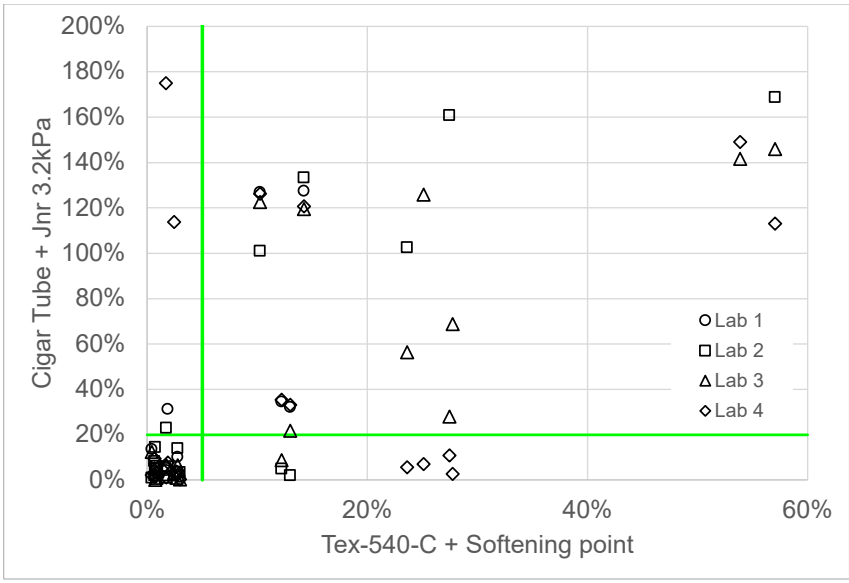


Figure 4.11. Comparison of Tex-540-C and Softening Point with ASTM D7173 and Jnr from MSCR Test at 3.2 kPa at high PG temperature or 64C for AC binders.

Figure 4.12 through Figure 4.15 compare the results from two replicates for the four parameters measured using the MSCR test. These comparisons also illustrate that the Elastic Recovery at 0.1 kPa parameter from the MSCR test is the most repeatable compared to other three parameters.

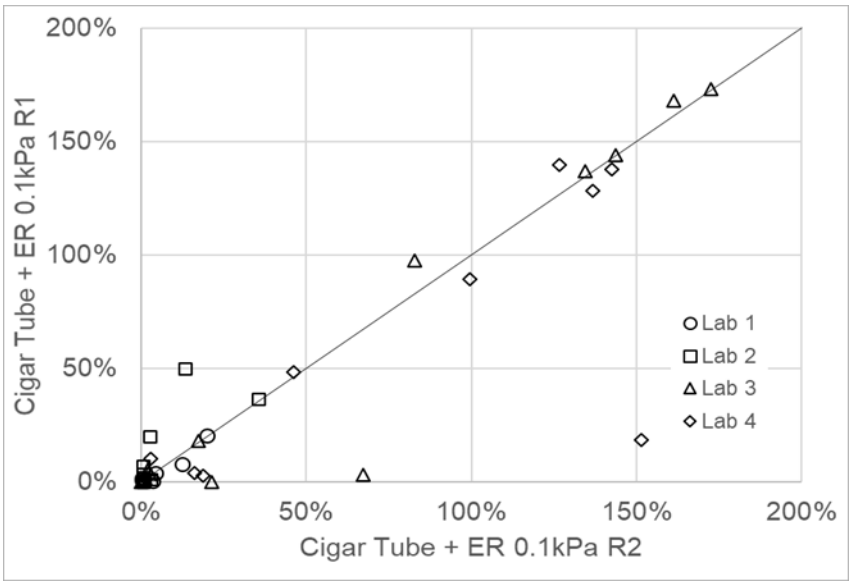


Figure 4.12. Comparison of separation measured using the two replicates based on Elastic Recovery from MSCR test at 0.1 kPa.

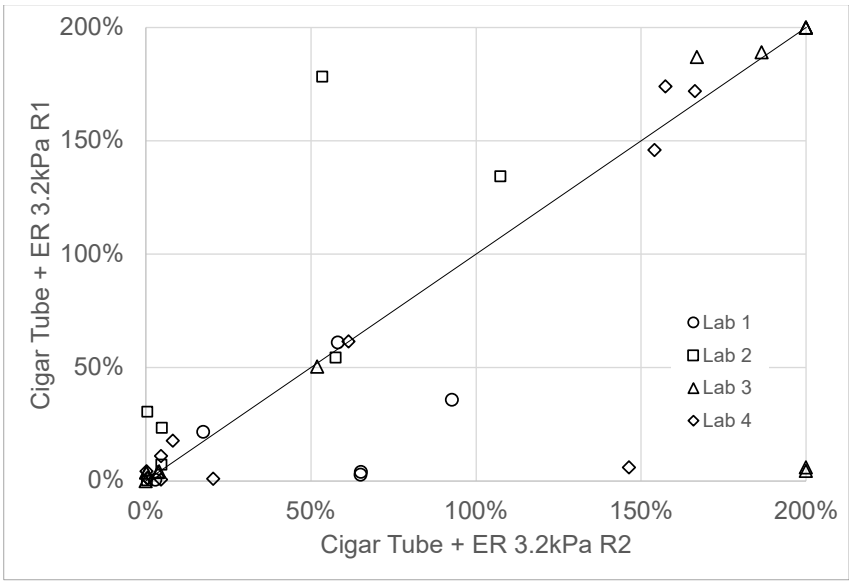


Figure 4.13. Comparison of separation measured using the two replicates based on Elastic Recovery from MSCR test at 3.2 kPa.

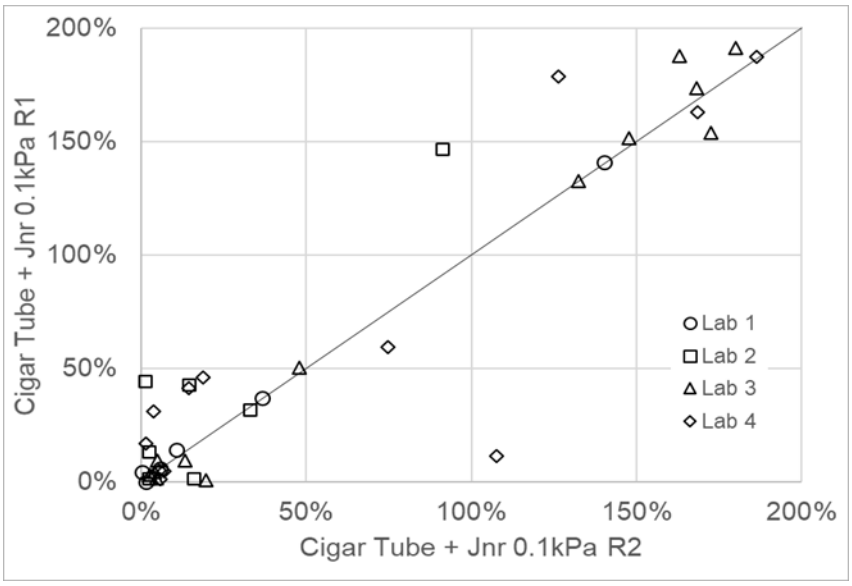


Figure 4.14. Comparison of separation measured using the two replicates based on Jnr from MSCR test at 0.1 kPa.

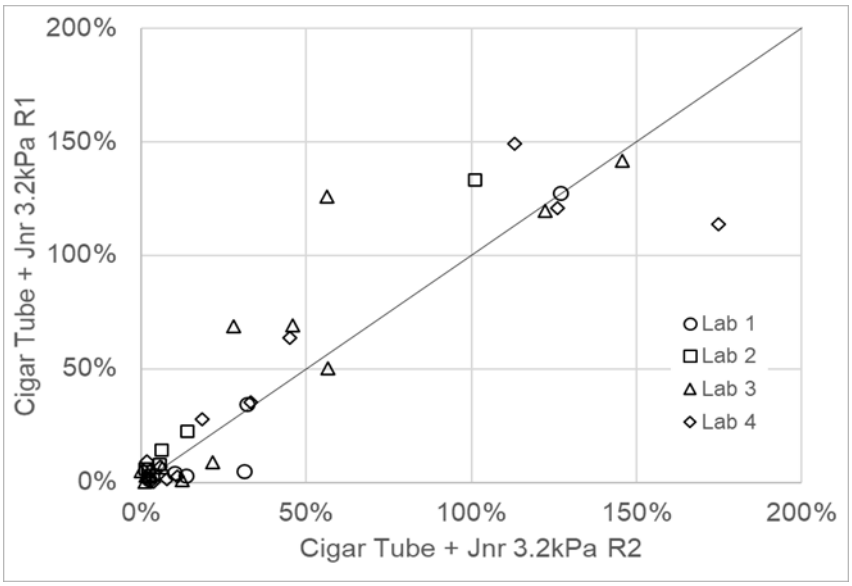


Figure 4.15. Comparison of separation measured using the two replicates based on Jnr from MSCR test at 3.2 kPa.

4.1.5. Conclusion and Recommendation

Overall, based on the data collected from this exercise, it is recommended to use ASTM D7173 for the separation test with G*/sin δ as the parameter to screen for whether separation has occurred. A percentage difference value of 10% can be used as a threshold for acceptance.

4.2. AASHTO T59 and T78 Emulsion and Cutback Recovery

4.2.1. Goal

The goal for developing this new test procedure, was stated in 3.2.1. This section describes the work performed to develop/modify a test procedure and method to establish residue recovery methods that can be used with cutbacks and emulsions..

4.2.2. Scope

The method is intended to be applied to the materials listed below in Table 4.4. The numbers included in the third column indicate the approximate testing load based on a 12-month analysis of the LIMS data).

Table 4.4 Emulsion and cutback residue recovery testing load based on LIMS data

TABLE	ITEM	TESTS/YR**
Table 4: Rapid curing cutback asphalt	RC-250	179
	RC-800	0
	RC-3000	0
Table 5: Medium curing cutback asphalt	MC-30	168
	MC-800	19
	MC-3000	10
	MC-250	0
Table 6: Special use cutback asphalt	SCM I	14
	MC-2400L	0
Table 7: Emulsified asphalt	HFRS-2	43
	MS-2	28
	SS-1	81
	SS-1H	82
	AES-300	0
Table 8: Cationic emulsified asphalt	CRS-2	202
	CMS-2	13
	CSS-1	37
	CSS-1H	336
	CRS-2H	0
	CMS-2S	0
Table 9: Polymer modified emulsified asphalt	HFRS-2P	52
	RS-1P	0
	AES-150P	0
	AES-300P	0
	AES-300S	0
	SS-1P	0
Table 10: Polymer modified cationic emulsified asphalt	CRS-1P	11
	CRS-2P	193
	CHRFS-2P	113
	CMS-1P	24
	CMS-2P	36

	CSS-1P	29
Table 11: Specialty emulsions	AE-P	195
	EAP&T	23
	PCE	20

** Based on LIMS data from 2020

4.2.3. Evaluation

4.2.3.1. Method

Several trial tests were conducted between the two research labs to develop method(s) that would result in repeatable and accurate results from both cutbacks and emulsions. These trials included variations with different substrate forms and materials (e.g. use of silicone molds, PAV trays, disposable aluminum weighing pans),temperatures used during vacuum recovery, and duration of recovery. After several different variations, two separate methods were finally recommended for residue recovery of cutbacks and emulsions. The next two subsections summarize these methods.

4.2.3.1.1. Cutback residue recovery

The vacuum oven was set to 140°C for cutbacks. The oven was preheated for a minimum of 24 hours to ensure temperature consistency and uniformity.

The cutback asphalt was stirred thoroughly for 1 minute using a spatula (Figure 4.16).

Figure 4.16. Cutback sample being stirred thoroughly.

The empty sample container[3] was weighed, and the mass (A) was recorded (Figure 4.17). Typical 4-oz size asphalt cans are recommended as a sample container, because the base of these

[3] An example of containers for cutback residue recovery: https://www.uline.com/Product/Detail/S-17906SIL/Retail-Boxes/Deep-Metal-Tins-Round-4-oz-Solid-Lid-Silver

containers is flat and the walls provide rigidity, which in turn ensures that a uniform film is formed.

Figure 4.17. Empty can for cutback residue recovery being weighed on scale.

The balance was zeroed, and a syringe was used to transfer the required amount of cutback from the sample source to the sample container. Typically, two containers were used to produce two replicate residue samples for each cutback sample. The mass poured in the sample container (B) was recorded (Figure 4.18). The sample amount is different for each viscosity grade of cutback to adjust for the binder content. The following sample amounts were used: for MC-30 use 2.70 g ± 0.05 g, and for RC-250 use 2.00 g ± 0.05 g. It is important to note here that these weights were based on a container diameter of 6 cm. If the diameter of the container being used is slightly larger or smaller, this mass can be adjusted accordingly to achieve similar film thickness.

Figure 4.18. Required amount of cutback sample being poured in the can.

The sample container was swirled to spread the cutback to get a uniform thickness of the cutback asphalt layer, as shown on Figure 4.19.

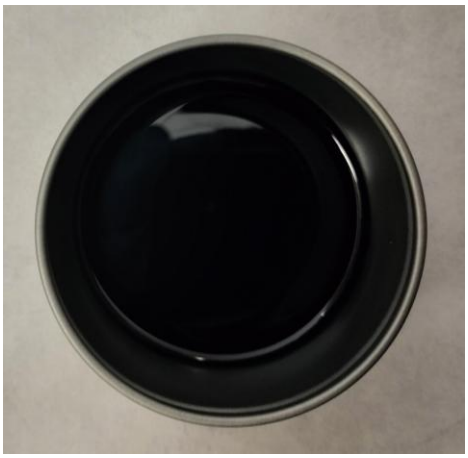


Figure 4.19. Cutback sample being spread in the can to get a uniform layer

Sample containers were placed in the vacuum oven (Figure 4.20) at 140°C ± 2°C, and a vacuum pressure of 90 mbar ± 10 mbar (absolute pressure) was applied for the specified time. For Rapid Curing Cutbacks, three hours of vacuum oven curing time was used, and for Medium Curing Cutbacks, four hours of vacuum oven curing time was used.

Figure 4.20. Cutback sample in vacuum oven[4] under the required pressure for residue recovery.

After the specified curing time under vacuum, the samples were removed from the oven and the sample containers were weighed immediately, as shown in

Figure 4.21, and the mass was recorded (C) to calculate the mass loss ((A+B-C)*100/B).

[4] An example of vacuum oven: https://www.acrossinternational.com/250c-ul-certified-1-9-cf-vacuum-oven-5-sided-heat-sst-tubing.html

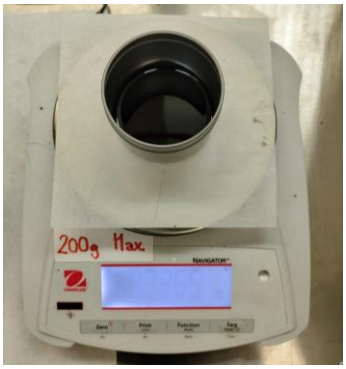


Figure 4.21. Cutback residue being weighed after the specified time in vacuum oven.

A clean spatula was used to scrape the residue from the can to create a DSR sample for performance testing (Figure 4.22).

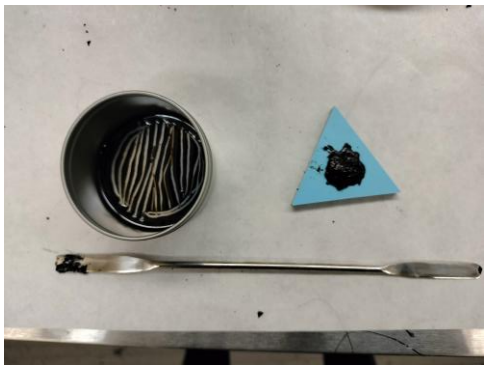


Figure 4.22. Scraping the cutback residue for testing in DSR.

4.2.3.1.2. Emulsion residue recovery

The vacuum oven was set to 70°C for asphalt emulsion. The oven was preheated for a minimum of 24 hours to ensure temperature consistency and uniformity.

The emulsion was stirred thoroughly for 1 minute using a wooden spatula as shown in Figure 4.23.

Figure 4.23. Emulsion sample being stirred thoroughly.

The sample container[5] (Figure 4.24) for emulsion residue recovery was weighed, and the mass (A) was recorded.

[5] An example of the container for emulsion residue recovery: https://www.mcmaster.com/17805T82/

Figure 4.24. Empty container for emulsion residue recovery being weighed on scale.

The balance was zeroed, and then a syringe was used to transfer 5.20 g ± 0.05 g of asphalt emulsion to the sample container. Typically, two replicate containers for each emulsion were used. The mass poured in the sample container was recorded (B) as shown in Figure 4.25.

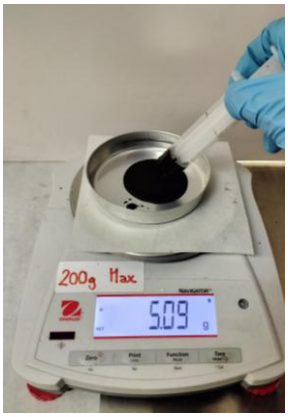


Figure 4.25. Required amount of emulsion sample being poured in the container.

The sample container was swirled to spread the emulsion in the mold to get a uniform thickness of the emulsion layer (Figure 4.26).

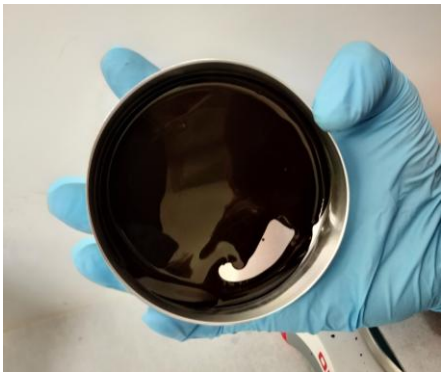


Figure 4.26. Emulsion sample being spread in the container to get a uniform layer.

Sample containers were placed in the oven at 70°C ± 2°C, and a vacuum pressure of 90 mbar ± 10 mbar (absolute pressure) was applied for two hours (Figure 4.27).

Figure 4.27. Emulsion sample in vacuum oven under the required pressure for residue recovery.

After two hours under vacuum, the samples were removed from the oven, and the containers were weighed immediately as shown in the Figure 4.28. The mass (C) was recorded to calculate the mass loss ((A+B-C)*100/B).

Figure 4.28. Emulsion residue being weighed after the specified time in vacuum oven.

A clean spatula was then used to scrape the emulsion residue from the container to a DSR sample for performance testing (Figure 4.29).

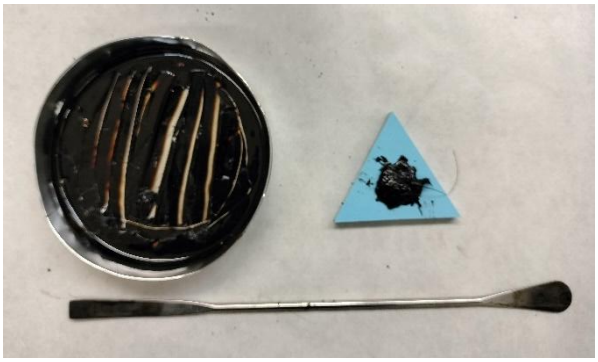


Figure 4.29. Scraping the emulsion residue for testing in DSR.

4.2.3.2. Materials and Tests

The following materials were used for evaluation. These materials cover cutbacks (RC and MC), and emulsions (slow-setting, rapid-setting, and high float). The materials were supplied by producers for the state of Texas.

- RC-250 x 3 suppliers
- MC-30 x 3 suppliers
- CRS-2P x 3 suppliers
- CHFRS-2P x 1 suppliers
- SS-1 x 3 suppliers

Dynamic shear rheometer (DSR) was used for performance testing on the residues obtained from cutbacks and emulsions. The residues were tested within 24 hours of obtaining the residues and preparing the DSR samples. Note that the same DSR test method was used both for cutback residues and emulsion residues. The DSR test is summarized below:

- Condition the sample at 64°C.
- Oscillate at 10% strain rate, 10 rad/s for ten cycles.
- Condition the sample at 25°C.
- Oscillate at 1% strain rate, 0.1 rad/s for ten cycles.

The parameter $G^*/\sin\delta$ from the last five cycles was averaged both at 64°C and 25°C. This parameter was used as a rheological indicator for the rheological properties of the cutback and emulsion residues. Note that the testing at 64°C using DSR is intended to serve as a rheological property index in lieu of the viscosity test requirement at high temperature (140°F) and the test at 25°C is intended to serve as a rheological index in lieu of the ductility or the penetration test requirement at intermediate temperature (77°F). The residue recovery procedure was developed based on the testing in two different labs on two different DSR brands. The results obtained are discussed in the subsequent section.

4.2.4. Results and discussion

4.2.4.1. Cutbacks

4.2.4.1.1. Testing.

Several trials were run to develop the procedure summarized in the previous section. Different containers, sample quantities, duration in vacuum oven, etc., were used to develop the current procedure. The next paragraphs discuss the results from the penultimate procedure and the latest procedure for the cutback residue recovery. Replicate data was not averaged and is shown as-is in these figures.

Figure 4.30 shows the percent mass loss during residue recovery of cutbacks between the two labs using the penultimate procedure. It can be observed from the figure that the repeatability of solvent between the two labs is very good. Figure 4.31 and Figure 4.32 show the G*/sin δ parameter at 64°C between the two labs and between the two replicates, respectively. It can be seen that except for one cutback, the data align well with the line of equality both between the labs and between the replicates, showing a good inter-lab and intra-lab repeatability using this procedure. However, this procedure was further refined to improve the repeatability.

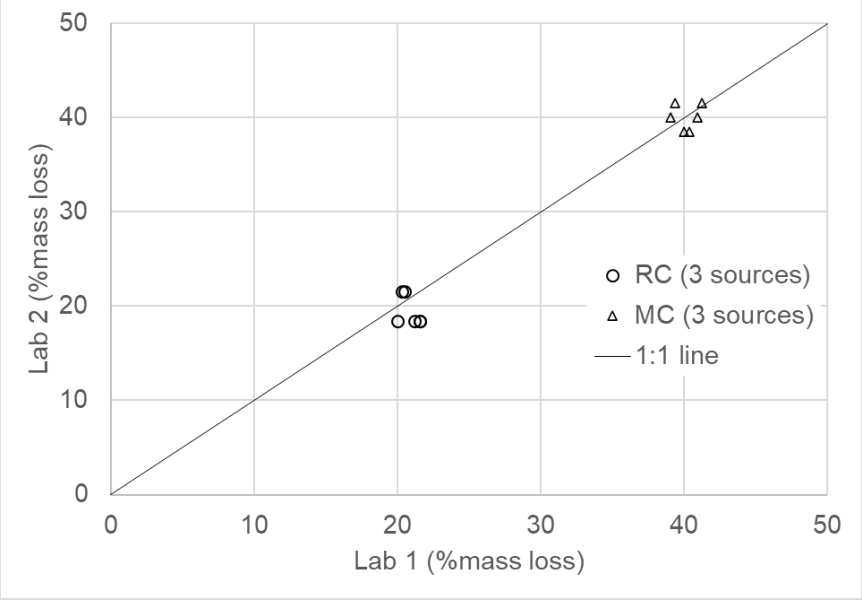


Figure 4.30. Comparison of mass loss during residue recovery between two labs (the line shown is 1:1 line for reference).

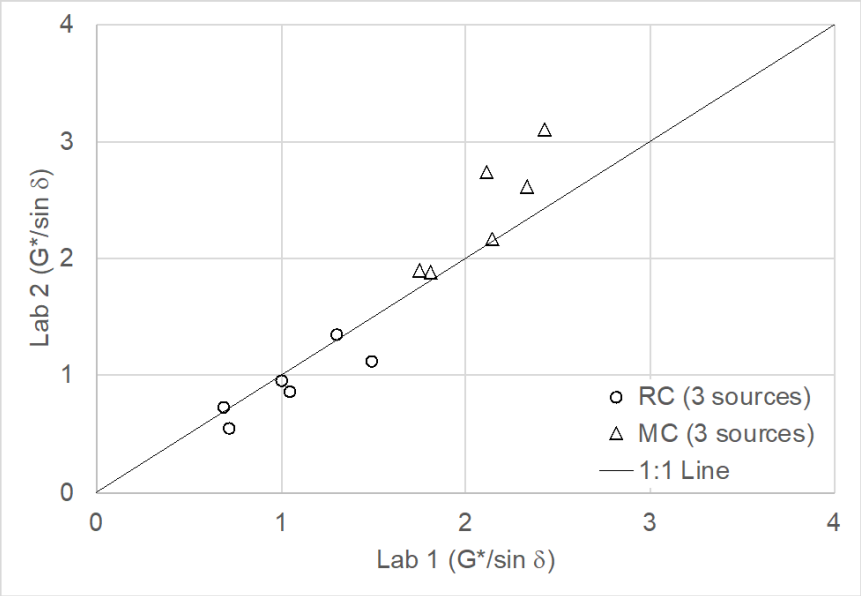


Figure 4.31. Comparison of G/sin δ parameter of the cutback residue between two labs (the line shown is 1:1 line for reference).*

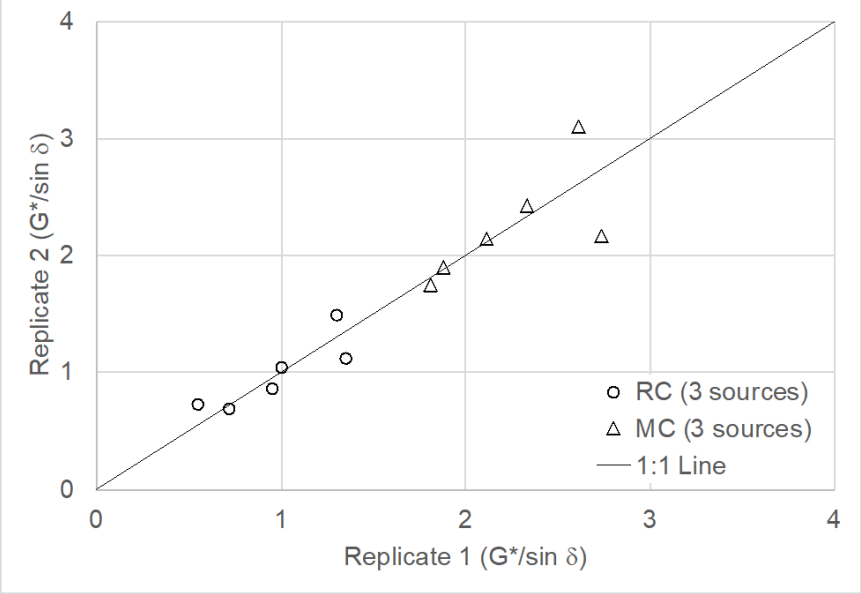


Figure 4.32. Comparison of G/sin δ parameter of the cutback residue between two replicates (the line shown is 1:1 line for reference).*

Two different cutbacks: MC 30 and RC 250 and a base binder for these cutbacks were obtained from a supplier to refine the procedure for cutback residue recovery. The containers for cutback residue recovery were changed, and the thickness of the cutback film was increased. Figure 4.33 and Figure 4.34 show the G*/sin δ parameter with the latest procedure (discussed in previous section) at 64°C and 25°C, respectively. It can be seen from these figures the repeatability between the two labs, as well as the repeatability between replicates, is very good at both high

temperature and intermediate temperature. It can also be observed that the residues are stiffer than the base which means that some of the lighter fractions were lost during the residue recovery.

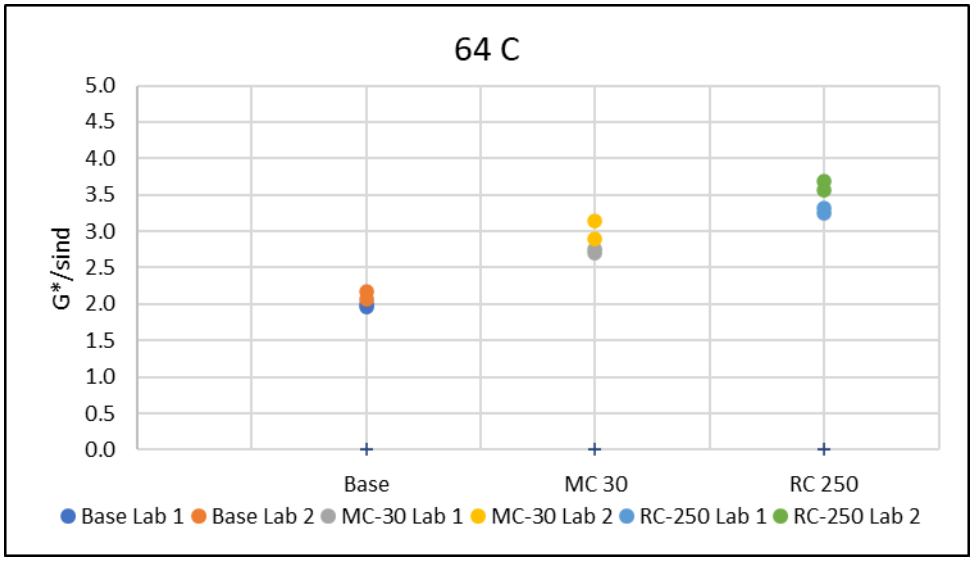


Figure 4.33. Comparison of G/sin δ parameter between two labs at 64°C for (i) base binder, (ii) MC 30 cutback, and (iii) RC 250 cutback.*

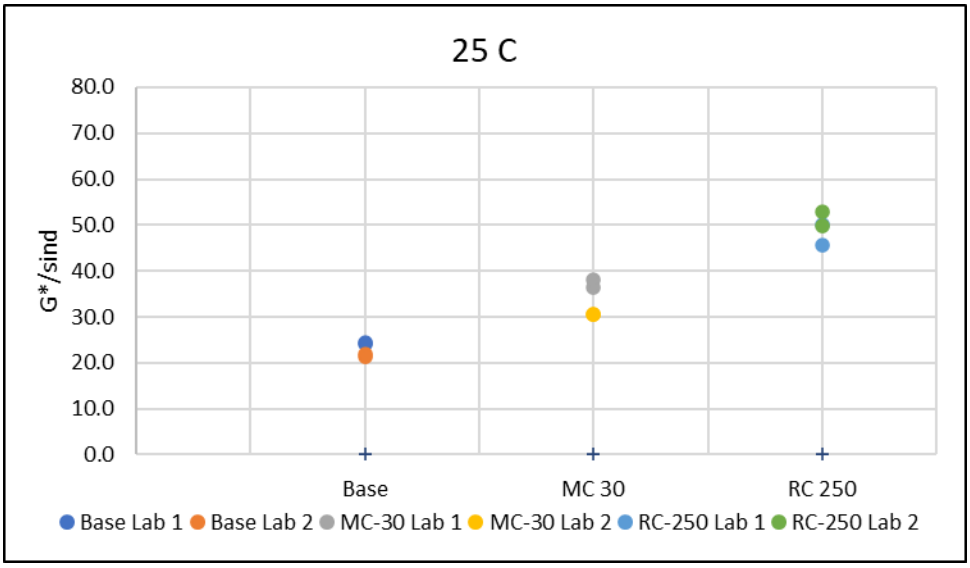


Figure 4.34. Comparison of G/sin δ parameter between two labs at 25°C for (i) base binder, (ii) MC 30 cutback, and (iii) RC 250 cutback.*

A round robin was conducted with several participating labs. RC-250 from two suppliers and MC-30 from two suppliers were used for this exercise. The results from the testing are shown in Figure 4.35 and Figure 4.36 below.

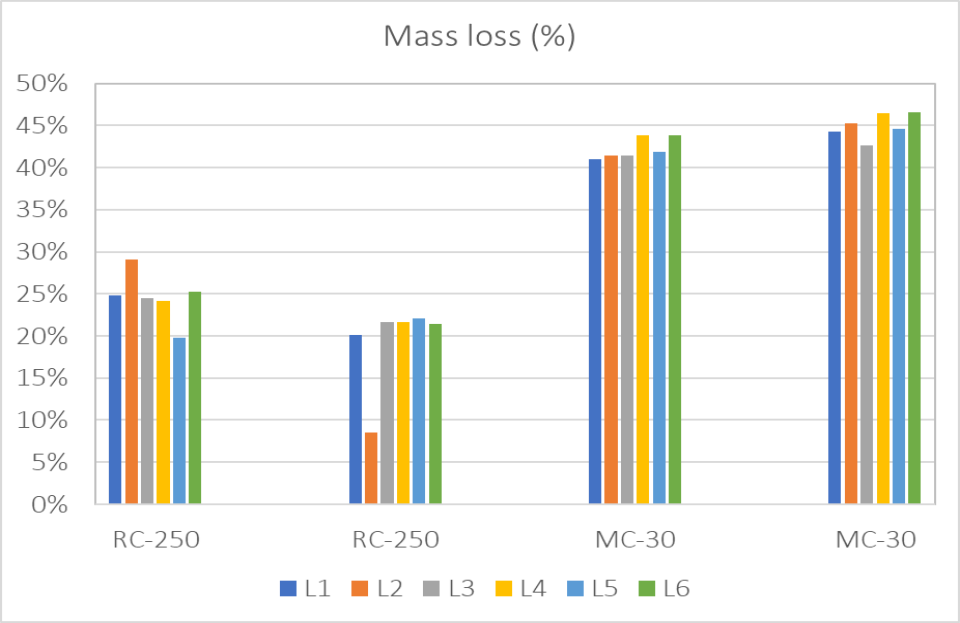


Figure 4.35. Mass loss data for the round-robin testing for cutback residue recovery.

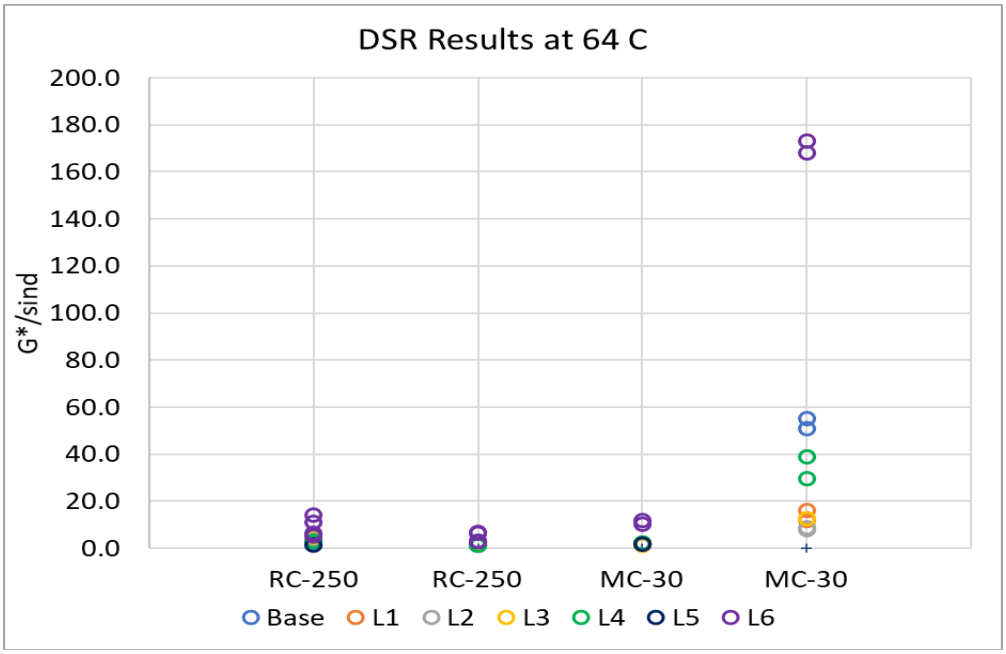


Figure 4.36. G/sin δ results from the round robin testing for cutback residue recovery.*

It can be observed from the above two figures that there is good repeatability between labs for mass loss during the recovery. A large variability was observed for one MC-30 due to the use of a very stiff base used to produce this cutback.

4.2.4.1.2. Compositional Analysis

As stated in 4.2.1, the vacuum recovery is not intended to provide a compositional analysis, as is the case with the distillation test used with cutbacks. The information about the distillate, to determine RC versus MC for instance, cannot be obtained with vacuum recovery. Several investigations were carried out to determine if this information could be easily obtained by another method. A summary of these investigations follows.

Gas Chromatograph/Mass Spectroscopy (GC/MS)

An approach to differentiate between RC and MC cutbacks was explored using a portable gas chromatography and mass spectrometry (GC/MS) system, designed to analyze chemical components. A new method was developed to detect and differentiate gasoline, kerosene, and diesel (the solvents usually used in the manufacture of cutback asphalts). By employing a vapor sampling method, the portable GC/MS test provides results within ten minutes. The Total Ion Chromatogram (TIC) from the test results is used to identify peak intensities of specific compounds unique to each solvent, based on their respective elution times. Solvent identification is then achieved by analyzing the intensity ratios of two selected compounds.

Following this method, a series of RC and MC cutback samples were tested. The raw intensity data from the TIC graph was processed through a series of steps to determine which of the three solvents were present.

The expected composition for different cure cutbacks is as follows:

- RC: Naphthalene/Gasoline
- MC: Kerosene
- SC: Diesel

Initial trials showed that the new method works effectively, although some RC cutbacks were incorrectly identified as kerosene. Further analysis revealed that the components and intensity of the misidentified RC cutbacks were very similar to kerosene, rather than gasoline, which was the expected result (Table 4.5).

Table 4.5. GCMS test results from RC250 and MC30 cutback distillation

Classification		GCMS Detection
RC250	2824	Kerosene
RC250	2928	Kerosene
RC250	3512	Gasoline
RC250	3553	Kerosene
RC250	3722	Gasoline
MC30	2449	Kerosene

MC30	2661	Kerosene
MC30	3973	Kerosene
MC30	3723	Kerosene

The Figure 4.37 shows the Total Ion Chromatograms of the RC250 cutback distillations. As shown, the composition of samples 2824, 2928, and 3553 are similar to one another but differ significantly from samples 3512 and 3722.

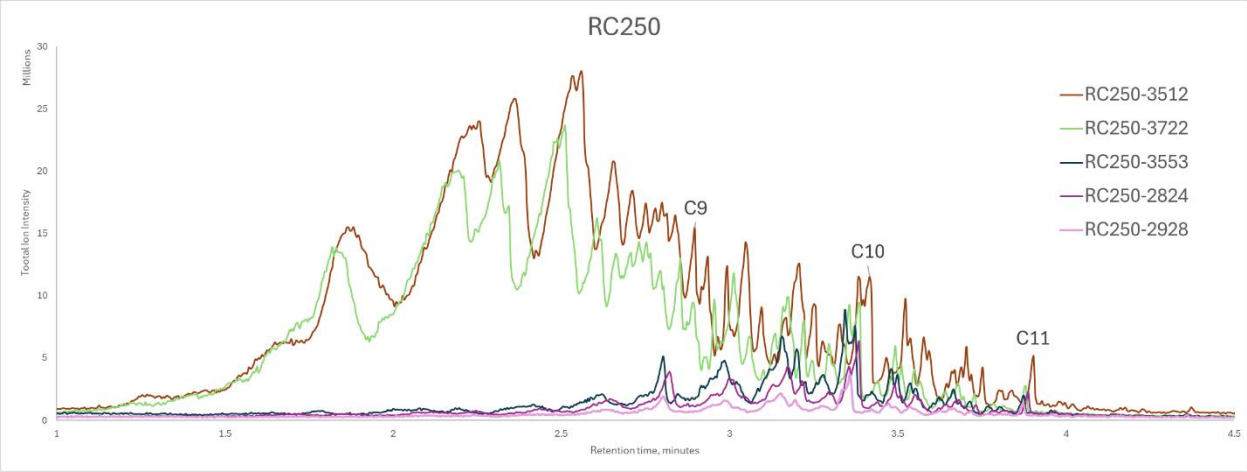


Figure 4.37. Total Ion Chromatograms of All RC250 Cutback Distillations Tested.

Gasoline/naphtha has a higher volatility than kerosene; therefore, it is expected to contain more of the lighter hydrocarbons. As a result, the chromatogram's intensity should be skewed towards the left compared to solvents that consist more of heavier hydrocarbons. Figure 4.38 compares the compositions of gasoline, kerosene, and the three misidentified RC250 cutbacks. The comparison reveals a strong similarity between the RC250 cutbacks and the composition of kerosene.

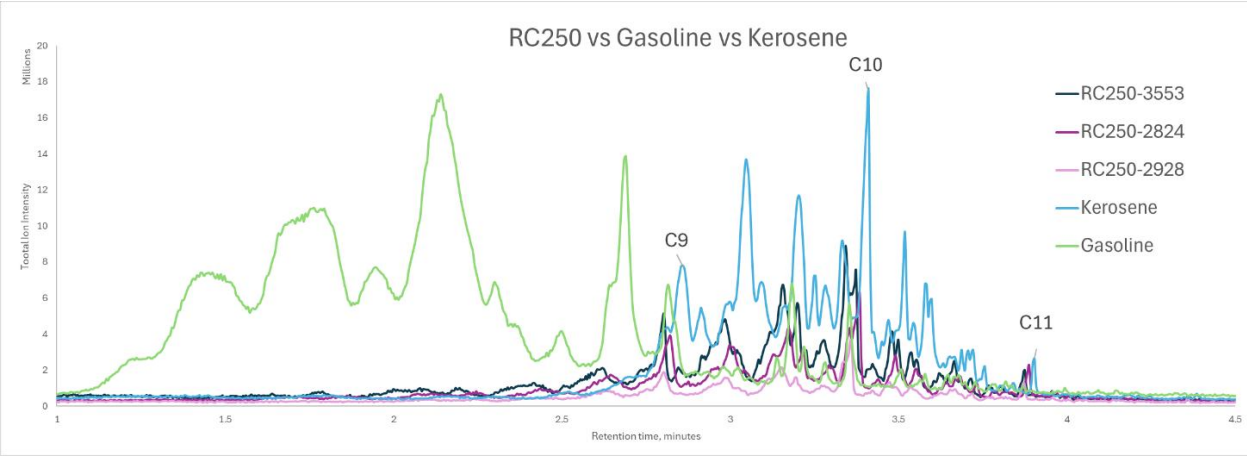


Figure 4.38. Comparing Compositions of RC250 Cutbacks, Gasoline and Kerosene.

Because of the misidentifications, this GC/MS work was not definitive in determining the type of cutback, so an additional correlation to use with the GC/MS was investigated.

Curing Index

Tex-517-C, "Test for Curing Index of Cutback Asphalts" is a TxDOT test procedure to calculate a curing index (CI). This CI is an indication of the speed of cure for a cutback. Lower CI values represent a faster curing cutback. The procedure uses data from the distillation test with "k" factors (constants depending on the boiling point of ranges of collected distillate) along with the volume of distillate collected at specific temperature intervals (Initial Boiling Point, 320°F, 347°F, 374°F, 437°F, 500°F, 600°F, and 680°F).

CI was investigated as data to use along with the GC/MS data to identify RC versus MC cutbacks.

The research team requested TxDOT data for the distillation of cutback samples contained in their Laboratory Information Management System (LIMS), and this information was delivered in a spreadsheet. On review, the data only contained distillation data at 437°F, 500°F, 600°F, and 680°F. This was not enough data to use Tex-517-C.

The team analyzed the data using a Surrogate CI that only used the data available in the TxDOT LIMS.

During the analysis, it appeared that the k-values in Tex-517-C for temperatures above 600°F were not correct. They did not fit the rest of the data. Including the two points in the test procedure that are above 600°F creates a best fit polynomial with an R2 of 0.6467, shown in Figure 4.39. If these two points are excluded the k-value data plots as a second order polynomial with an R2 of 0.9878 as shown in Figure 4.40. It was decided that the k-values above 600°F were not correct, and the polynomial fit determined without them would be used to estimate new k-values that fit the rest of the data.

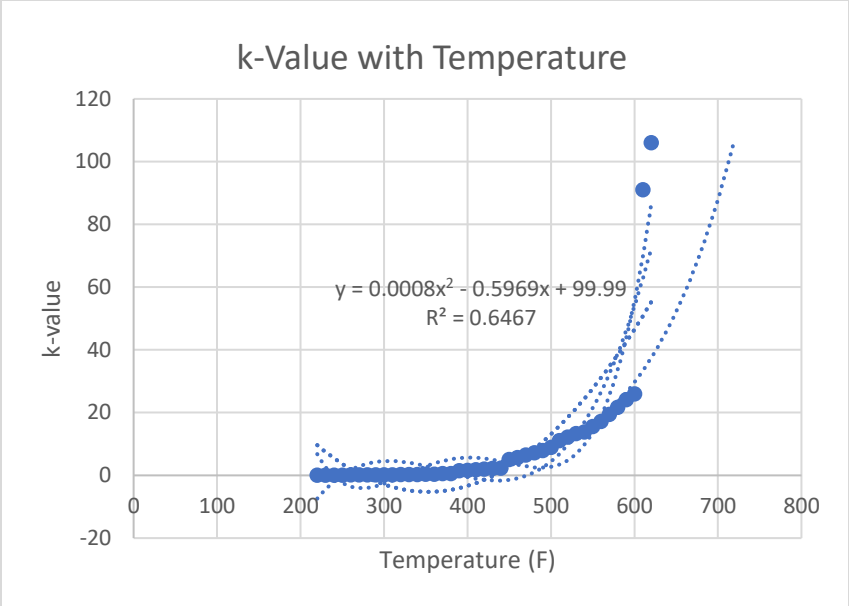


Figure 4.39. k-Values with Temperature with all k-values used.

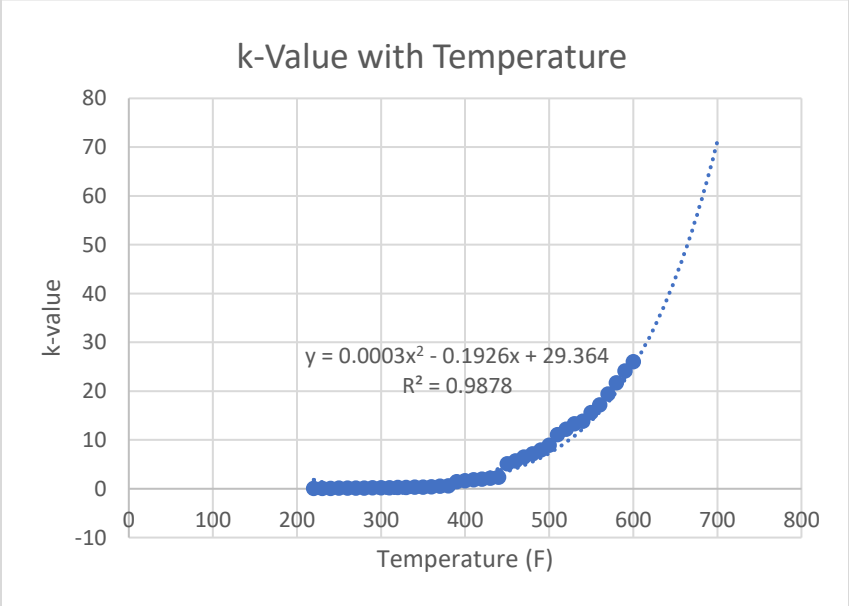


Figure 4.40. k-Values with Temperature, excluding points above 600°F.

The k-values (including estimated values for temperatures above 600°F) were used with the truncated TxDOT data from LIMS to calculate the Surrogate CI. These are summarized in Table 4.5.

Table 4.5. Surrogate CI by Cutback Grade

	Surrogate CI		
Grade	**Min**	**Avg**	**Max**

RC-250	101	217	335
MC-30	397	645	931
MC-800	223	439	568
MC-3000	360	367	381
SCM-1	402	493	640

If one compares the CI within a grade, higher CIs belong to cutbacks with higher amounts of solvent. While this holds for the same grade on average, the spreads from high to low overlap to make an individual test not to distinguish a grade. Also, across grades show the same overlap that precludes making a distinction between RC and MC. The maximum CI of the RC-250 is 335, and the minimum CI of MC-800 is 223.

This work does not result in a measure that can be used with GC/MS to distinguish RC versus MC. Additionally, if one relied only on the vacuum recovery, the distillation data would not be available anyway.

Conclusions

While this exercise did not result in a measure to be used to determine RC versus MC cutbacks, additional review of the vacuum recovery test itself, along with testing of the residue recovered from that procedure offers a solution.

The vacuum oven procedure uses different time durations at 140°C for recovery, RC for 3-hours and MC for 4-hours. These times were determined to by experimentation as required to remove the solvent of different volatiles.

The most probable problems with cutbacks could include:

- Cutback Contamination (almost all contamination would be with higher boiling materials, with diesel or hydraulic fluid being the most common),
- Sample Mislabeling (RC is really an MC or MC is really an RC), or
- Cutbacks are Partially cured out (old sample of mistreated sample).

For these situations,

- Contamination would result in a cutback residue that would be much softer than expected.
- Sample mislabeling would result in residue either too stiff or much softer than expected.

- Partial curing would not result in too soft or hard residue, but would result in a cutback that would not meet the cutback viscosity requirements.

Thus, the most common ways for cutbacks to exhibit problems will produce test results that raise suspicion. Since the research team does not recommend deleting the AASHTO T78 procedure, but to have it available for forensic analysis or referee testing, cutback samples that raise suspicion can be evaluated using AASHTO T78 to verify that there is a problem.

4.2.4.2. Emulsions

Similar to the development of cutback residue recovery procedure, development of emulsion residue recovery procedure also required many trial procedures. Variables, such as temperature, duration in vacuum oven, and sample containers, were evaluated during the development of the test procedure. The following paragraphs discuss the results from the penultimate test procedure and the latest test procedure. Replicate data was not averaged and is shown as-is in the figures. Note that two research labs participated in the testing.

Figure 4.41 shows the comparison of G*/sin δ parameter between the two participating labs. It can be observed that the values lie very close to the line of equality showing good repeatability between two labs. Recall that the two labs had different brands of vacuum ovens and DSRs.

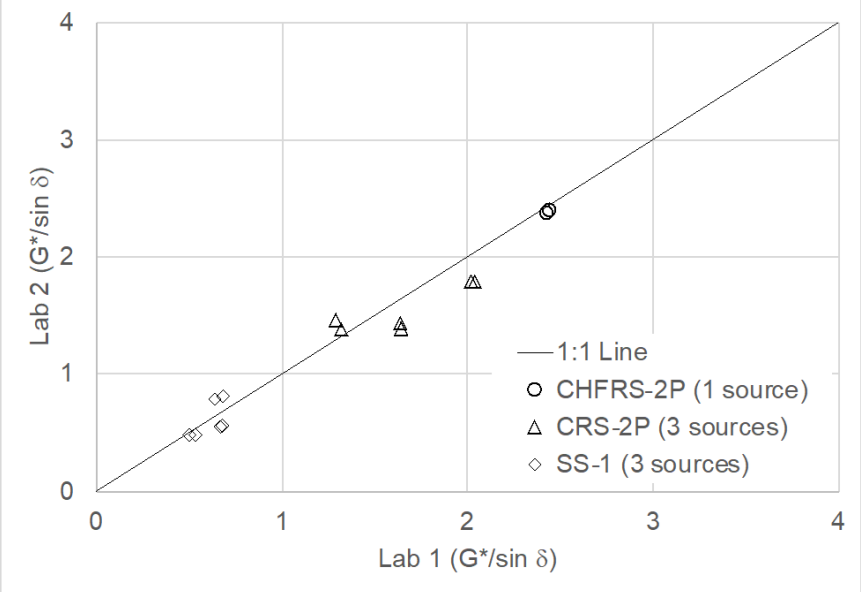


Figure 4.41. Comparison of G/sin δ parameter of the emulsion residue between two labs (the line shown is 1:1 line for reference).*

Figure 4.42 compares the G*/sin δ parameter between two replicates. The values are very close to the line of equality showing a good repeatability within the lab with same operator. These results are based on the penultimate procedure in which silicone containers were used as samples containers. However, in case of some emulsions it was observed that the emulsions do not spread

uniformly on silicone containers due to surface tension. This procedure was modified by using aluminum containers.

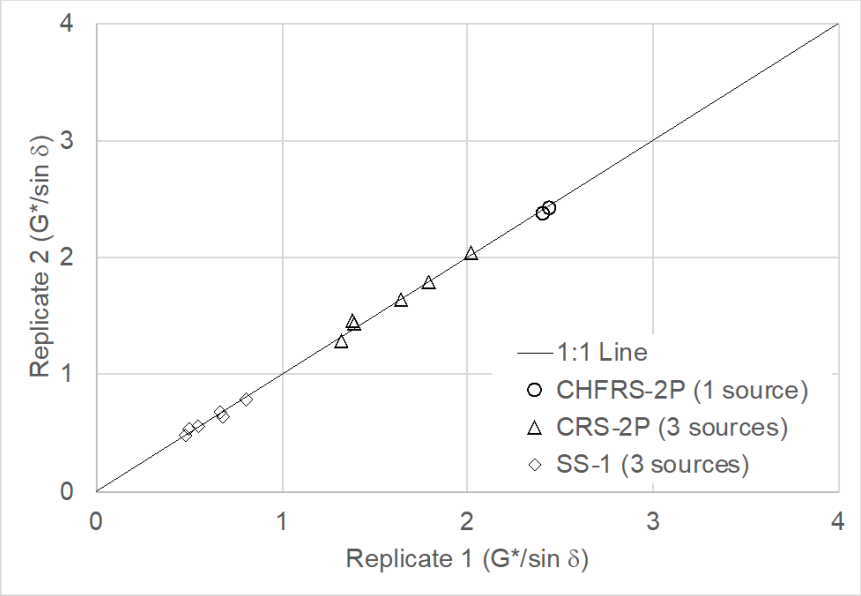


Figure 4.42. Comparison of G/sin δ parameter of the emulsion residue between two replicates (the line shown is 1:1 line for reference).*

The results from the latest procedure using aluminum containers are shown in Figure 4.43 to Figure 4.47. Figure 4.43 shows the mass loss during the emulsion residue recovery between the labs. The results are very close to the line of equality showing a good repeatability in mass loss between the two labs. Figure 4.44 and Figure 4.45 show repeatability in G*/sin δ parameter at high temperature (64°C) in between the two labs and within the lab, respectively. Results for all three emulsions are close to line of equality in both plots showing both good repeatability within the lab as well as between the labs at high temperature. Figure 4.46 and Figure 4.47 show the repeatability of G*/sin δ parameter at intermediate temperature (25°C) in between the two labs and within the lab, respectively. Data in both the plots is close to line of equality showing good inter-lab and intra-lab repeatability at intermediate temperature.

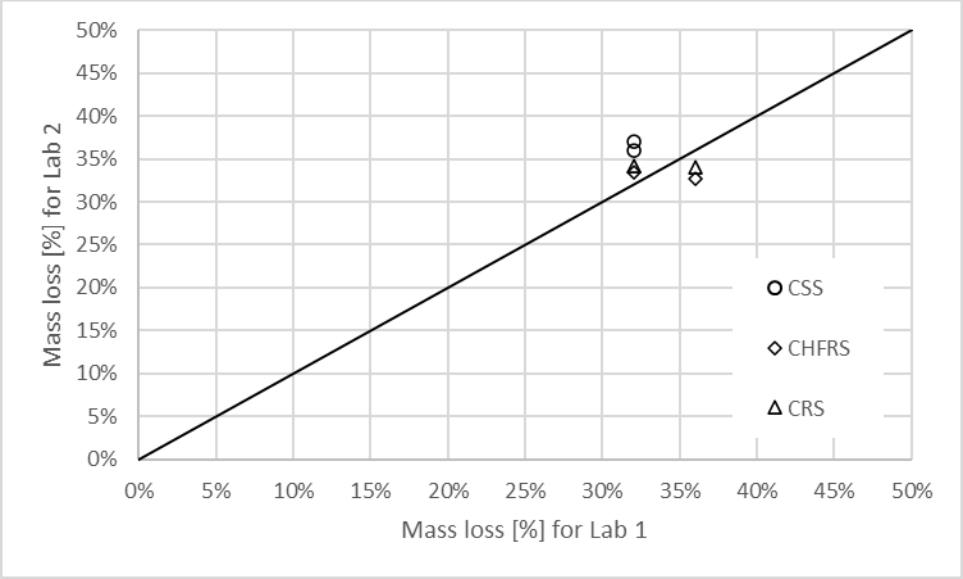


Figure 4.43. Comparison of mass loss percent during emulsion residue recovery between two labs (the line shown is 1:1 line for reference).

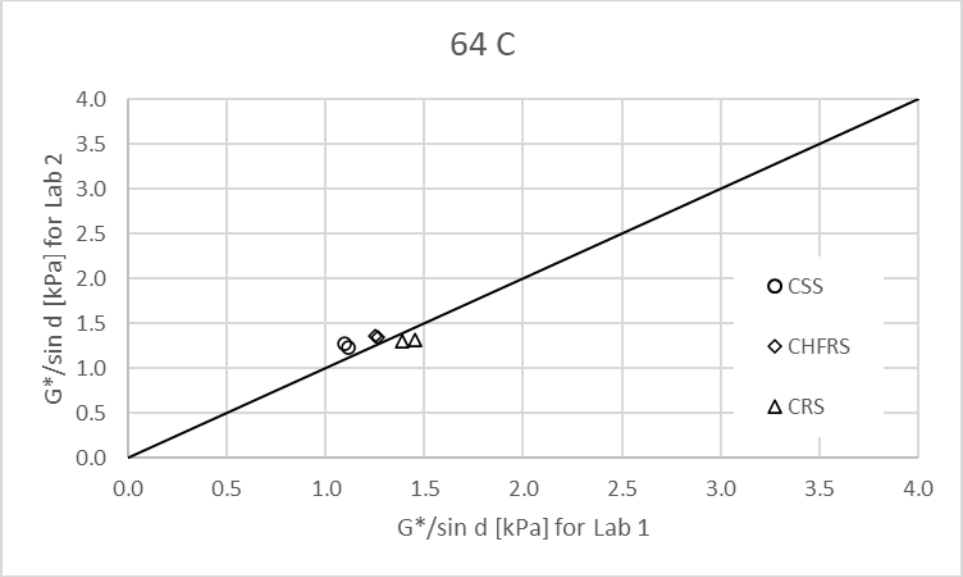


Figure 4.44. Comparison of G/sin δ parameter of the emulsion residue between two labs at high temperature (the line shown is 1:1 line for reference).*

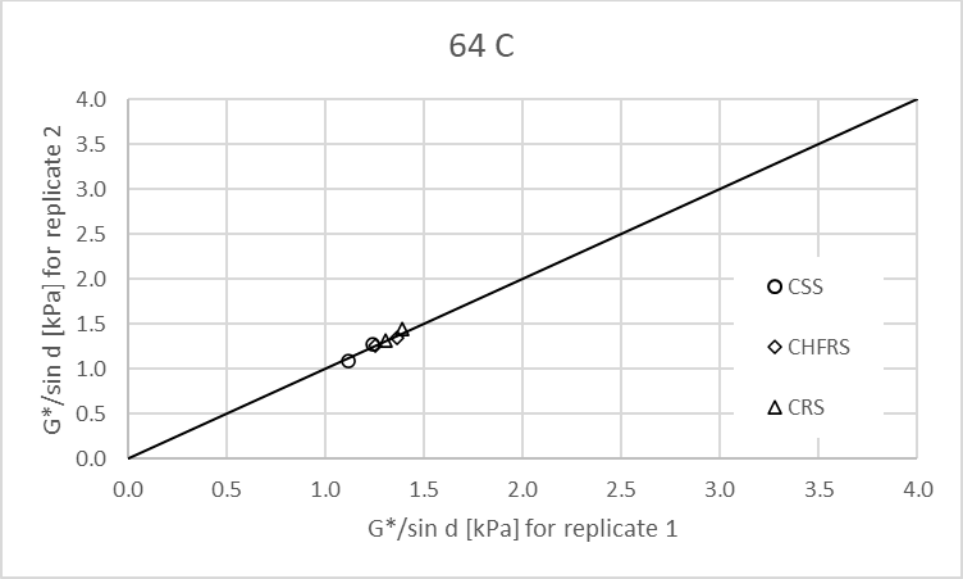


Figure 4.45. Comparison of G/sin δ parameter of the emulsion residue between two replicates at high temperature (the line shown is 1:1 line for reference).*

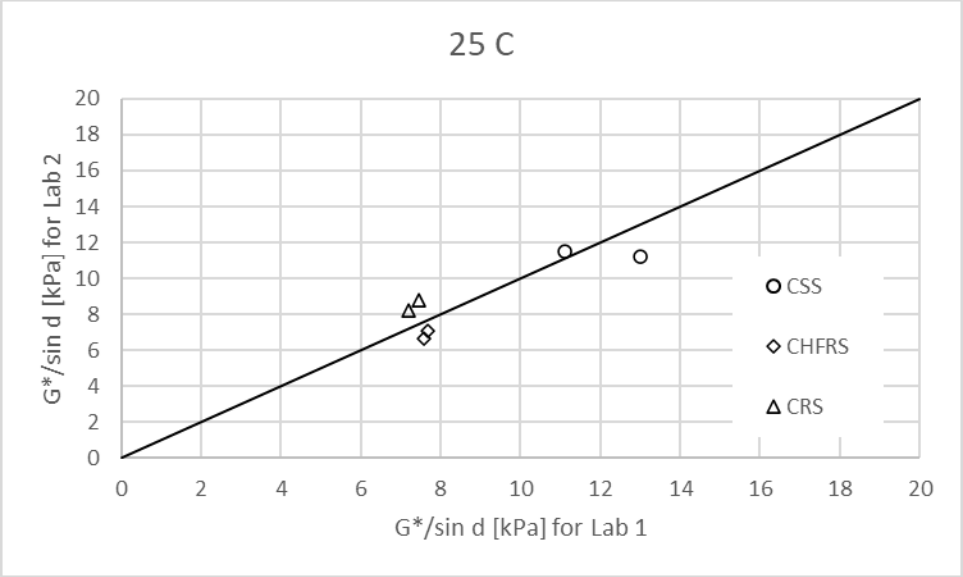


Figure 4.46. Comparison of G/sin δ parameter of the emulsion residue between two labs at intermediate temperature (the line shown is 1:1 line for reference).*

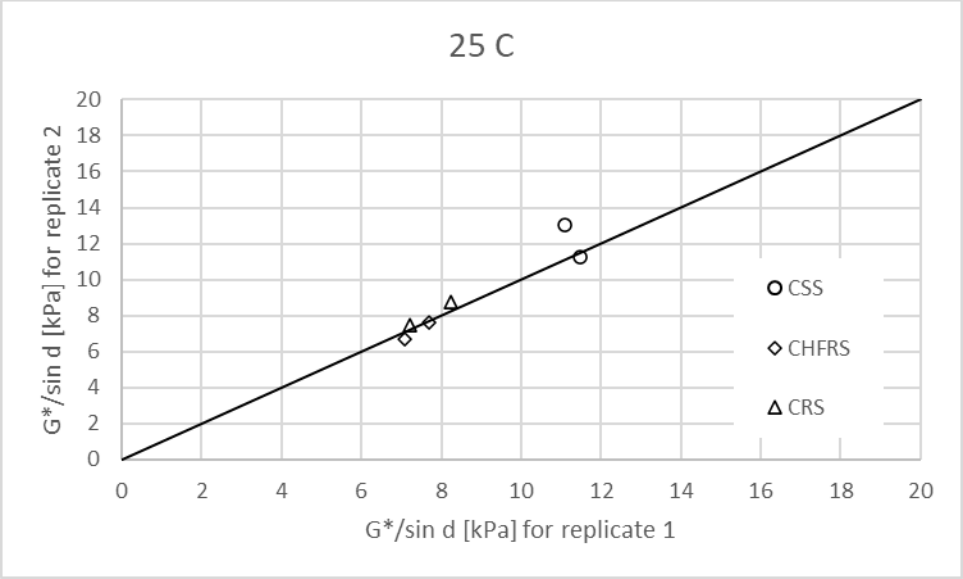


Figure 4.47. Comparison of G/sin δ parameter of the emulsion residue between two replicates at intermediate temperature (the line shown is 1:1 line for reference).*

A round-robin testing with industry participation is being conducted with CRS-2P from two suppliers and HFRS-2P from one supplier. The results obtained from this round robin testing is shown in the Figure 4.48 and Figure 4.49 below.

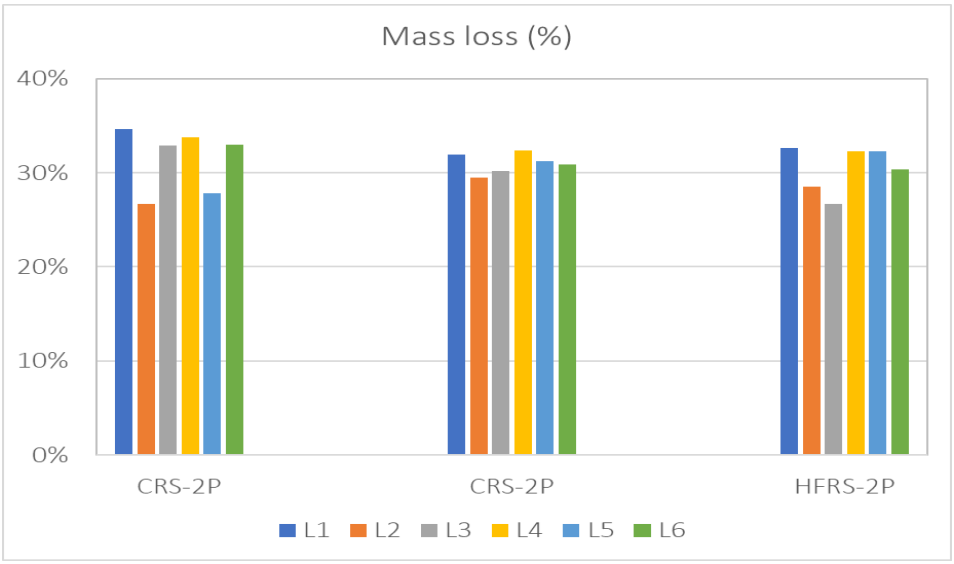


Figure 4.48. Mass data for the round-robin testing for emulsion residue recovery.

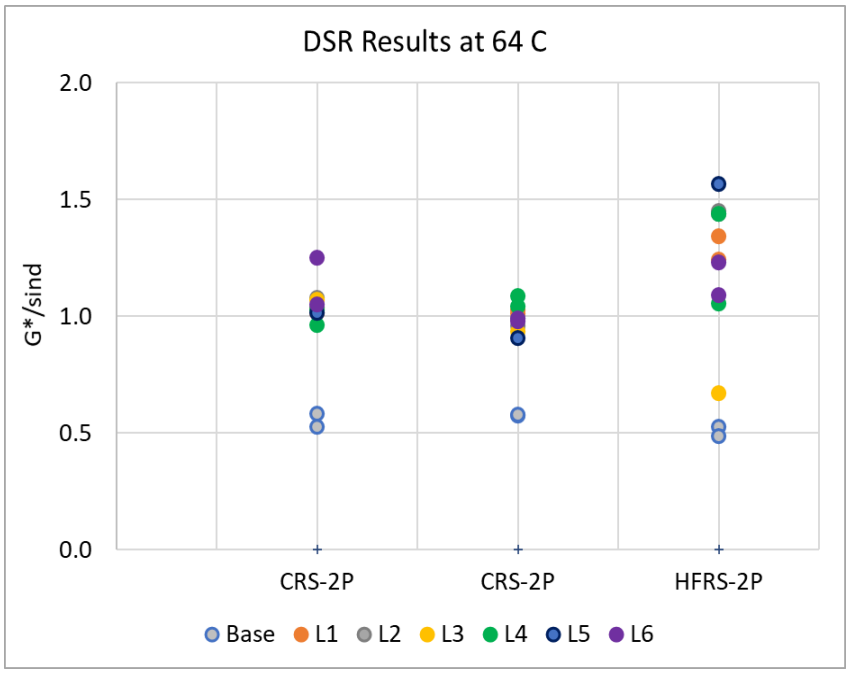


Figure 4.49. G/sin δ results from the round robin testing for emulsion residue recovery.*

A good repeatability for mass loss data can be observed between the six labs. Also, a good repeatability can be seen for the DSR G*/sin δ parameter. The variability in the G*/sin δ parameter was observed to be more in the case of HFRS-2P as compared to CRS-2P. Note that the base binder was softer than the recovered binder. In this case, according to the producer, the polymer was introduced during emulsification and is therefore present in the recovered binder, but not in the base binder.

4.2.5. Conclusion

Two separate test procedures were developed for the residue recovery of cutbacks and emulsions. The data collected so far shows a good repeatability within the lab as well as between different labs at both high and intermediate temperatures. A round robin was conducted with several participating labs from the industry. Results from the round robin show good repeatability between the labs.

4.3. AASHTO T201 Kinematic Viscosity of Asphalts

4.3.1. Goal

The main goals of this exercise were to:

- Replace T201 with rotational viscometer. This test is currently being performed at 140°F on RC, MC, and special-use cutbacks (Tables 4, 5, 6), as well as rejuvenating agents

(Table 10) and Residue of specialty emulsions (Table 11). The test is also being performed at 275°F on recycling agents and emulsified recycling agents (Table 12).

  - Some adjustment to the spec limit may be required to convert kinematic to absolute viscosity.
  - Some methods to measure this in the field are also being considered.

- Replace T201 (kinematic viscosity using a viscometer tube) for residue of specialty emulsions at 140°F (Table 11) with a DSR parameter.
  - Tests on residue also tie into a more efficient residue recovery process that yields a smaller sample size.
  - The shift to replacing a viscosity-based consistency measure with DSR is consistent with multiple instances throughout Item 300.
  - Table 3 already sets a precedent for the use of DSR and G*/sin δ.

4.3.2. Scope

The method is intended to be applied to the materials in Table 4.6 below. The numbers included in the third column indicate the approximate testing load based on a 12-month analysis of the LIMS data).

Table 4.6 Kinematic viscosity testing load based on LIMS data

TABLE	ITEM	TESTS/YR**
Table 4: MC cutbacks (140°F)	RC-250	179
Table 5: MC cutbacks (140°F)	MC-30	168
	MC-800	19
	MC-3000	10
Table 6: Special-use cutbacks (140°F)	SCM I	14
Table 10: PM Cationic emulsions (140°F)	CRS-2P	192
	CHFRS-2P	112
	CMS-1P	1
	CMS-2P	1
Table 11: Specialty emulsions (140°F)	PCE	21

** Based on LIMS data from 2020

4.3.3. Evaluation Method

4.3.3.1. Methods

AASHTO T201

AASHTO T316

Calculate Density to convert the kinematic viscosity to absolute viscosity

4.3.3.2. Materials

Materials used for data collection were as follows:

- RC-250 x 3 different sources
- SCM I x 1 different sources
- MC-30 x 3 different sources

4.3.3.3. Kinematic Viscosity Test

- Test was conducted at 140°F.
- Tubes were placed in the bath for 20 minutes, then asphalt was poured into the tubes, and the test was started after 20 minutes.
- Time of flow was recorded, and viscosity was calculated by multiplying the recorded time by the tube constant.

4.3.3.4. Rotational Viscosity Test

- Test is conducted at 60°C.
- Samples were placed in the viscometer for ten minutes, then the spindle was turned on for an additional ten minutes. This procedure was subsequently refined.
- Three readings were taken, and the average is reported.

4.3.4. Results and Discussion

Two laboratories participated to test the cutbacks with the rotational viscometer. Figure 4.50 presents a comparison of rotational viscosity results obtained between the two labs. It can be seen that the data aligns very well with the line of equality showing very good repeatability between two labs. Each group of data in the form of clusters represents a different type of cutback.

The correlation observed between the results from these two tests provides preliminary evidence to consider replacing tube viscometers with rotational viscometers for cutback materials, without applying any significant changes to the current specifications.

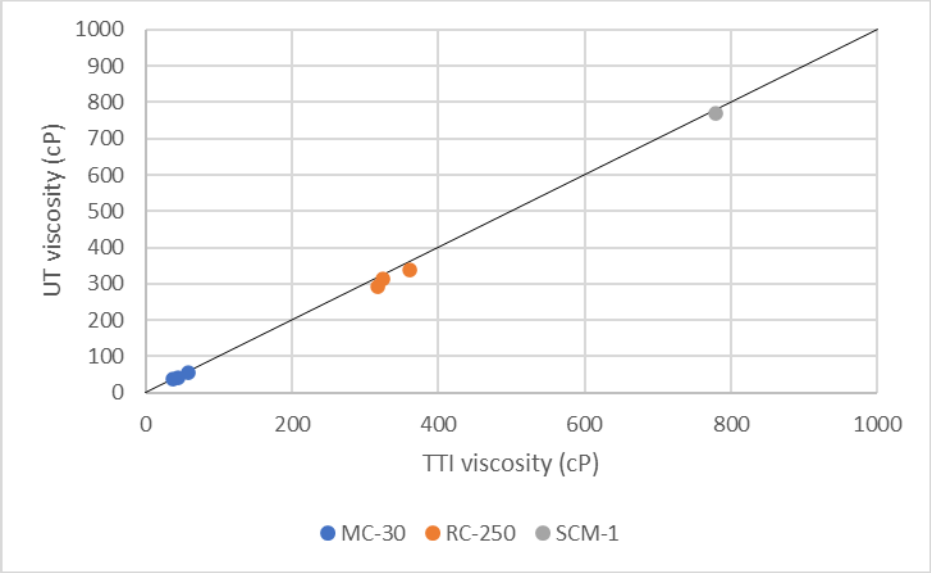


Figure 4.50 Comparison of rotational viscometer measurements between the two labs.

Figure 4.51 and Figure 4.52 presents the comparison of rotational viscosity and kinematic viscosity from the two labs, respectively. It can be seen that data aligns very close to the line of equality showing the limits used for kinematic viscosity can be used with rotational viscosity measurements.

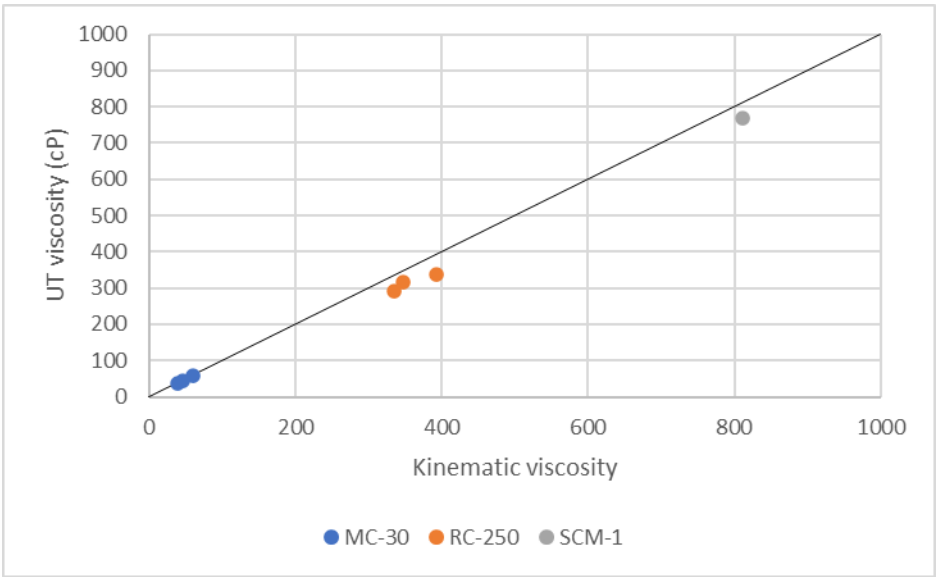


Figure 4.51. Comparison of rotational viscometer measurements from lab at UT with kinematic viscosity.

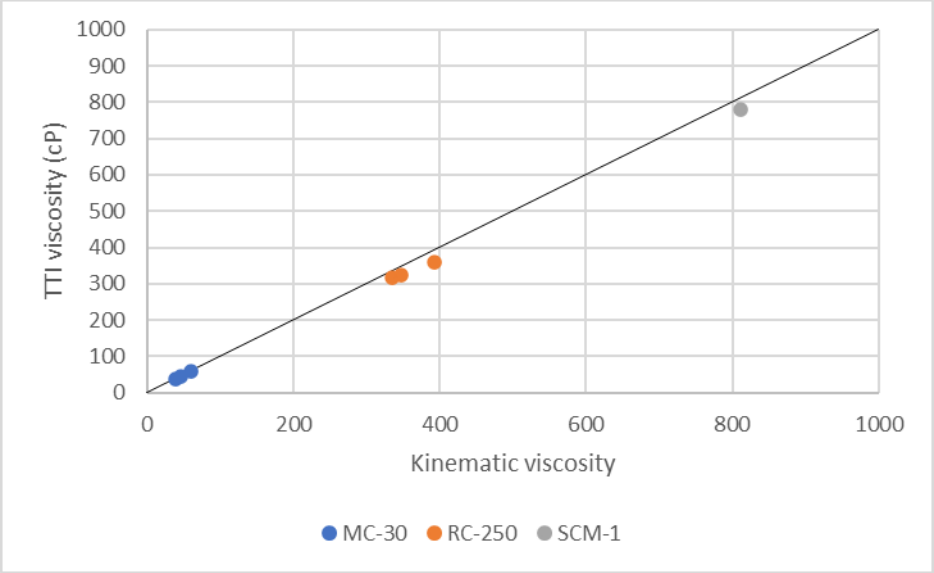


Figure 4.52. Comparison of rotational viscometer measurements from lab at TTI with kinematic viscosity.

4.3.5. Conclusion

Data suggests that for cutback materials, the rotational viscometer can be used in lieu of the tube viscometers with only minor, if any, revisions to the current parameters or specification limits.

4.4. AASHTO T72 Saybolt viscosity

4.4.1. Goal

The main goal of this exercise was to replace T72 with rotational viscometer. This test is currently being performed at 77°F or 122°F on all type of emulsions (Tables 7 to 11), and at 77°F on recycling agents and emulsified recycling agents.

4.4.2. Scope

The method is intended to be applied to the materials as listed in Table 4.7 below. The numbers included in the last column indicate the approximate testing load based on a 12-month analysis of the LIMS data).

Table 4.7 Saybolt viscosity testing load based on LIMS data

TABLE		ITEM	TESTS/YR
Table 7: Emulsions	77°F	SS-1	81
		SS-1H	83
	122°F	HFRS-2	43
		MS-2	28
Table 8: Cationic emulsions	77°F	CSS-1	37
		CSS-1H	333
	122°F	CRS-2	198

		CMS-2	13
		CSS-1H	1
Table 9: PM emulsions	122°F	HFRS-2P	52
Table 10: PM Cationic emulsions	77°F	CMS-1P	24
		CSS-1P	29
	122°F	CRS-1P	11
		CRS-2P	192
		CHFRS-2P	113
		CMS-2P	35
Table 11: Specialty emulsions	77°F	EAP&T	1
		PCE	17
	122°F	AEP	195

4.4.3. Evaluation Method

4.4.3.1. Method

AASHTO T72

ASTM D7226

4.4.3.2. Materials

Materials used for data collection were as follows:

- CSS-1H x 2 different sources
- SS-1 x 2 different sources
- CRS-2P x 3 different sources
- CRS-2 x 3 different sources
- HFRS-2P x 2 different sources

4.4.3.3. Saybolt Viscosity Test

Test is conducted at 77°F (25°C) or 122°F (50°C) depending on the grade.

4.4.3.4. Rotational Viscosity Test

Test is conducted at 60°C. The spindle was lowered for ten minutes in the sample; then the spindle was turned on for an additional ten minutes; and finally, the average of three readings was recorded.

4.4.4. Results and Discussion

Two labs participated in the testing. Saybolt viscometer results are compared with the rotational viscometer results in Figure 4.53 and Figure 4.54. It can be seen from these figures that the Saybolt viscosity test itself is very variable for most of the rapid setting emulsions. Hence, it was not possible to obtain a linear correlation between the two tests.

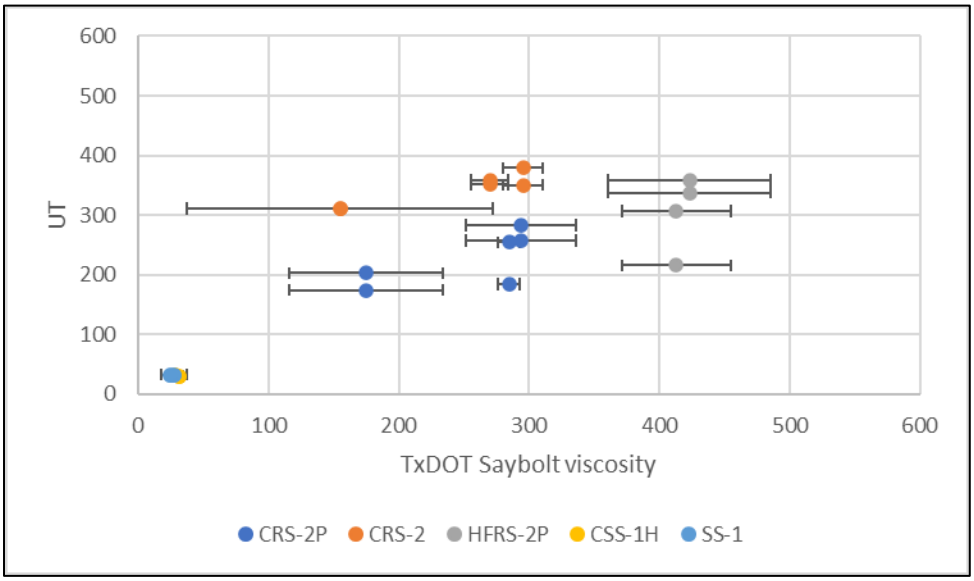


Figure 4.53. Comparison of rotational viscosity data obtained from UT lab with the Saybolt viscosity.

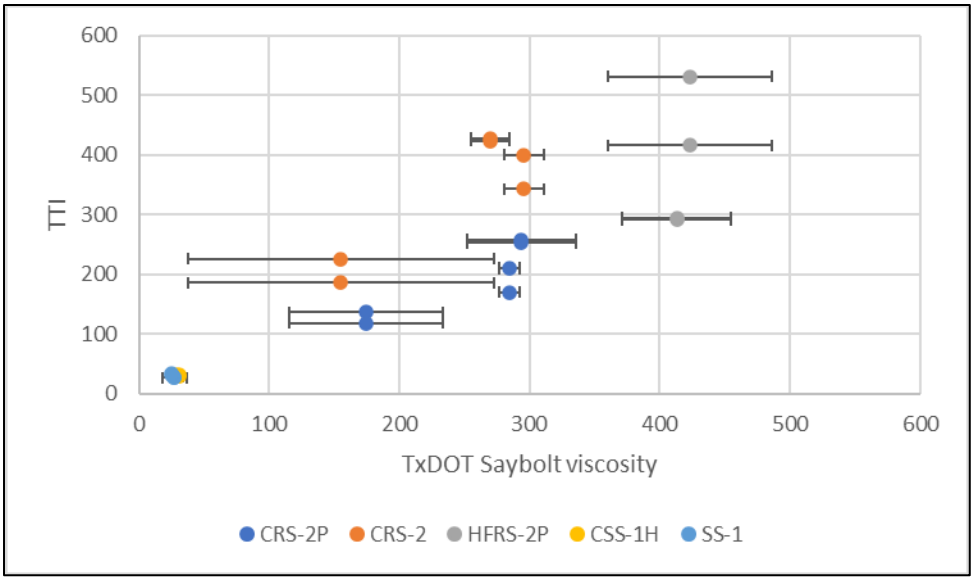


Figure 4.54. Comparison of rotational viscosity data obtained from TTI lab with the Saybolt viscosity.

Figure 4.55 and Figure 4.56 show the comparison of rotational viscosity data between two labs and between two replicates, respectively. A good repeatability can be observed with this new test

both between labs and between the replicates. Average d2s% between labs for Saybolt viscosity test was 28% with a minimum d2s% of 5% and a maximum d2s% of 86% for the material used in this exercise. On the other hand, the average d2s% for the rotational viscometer test on the same material was 16% with a minimum d2s% of 0% and maximum d2s% of 41%. This shows the repeatability improves significantly with the rotational viscosity test method.

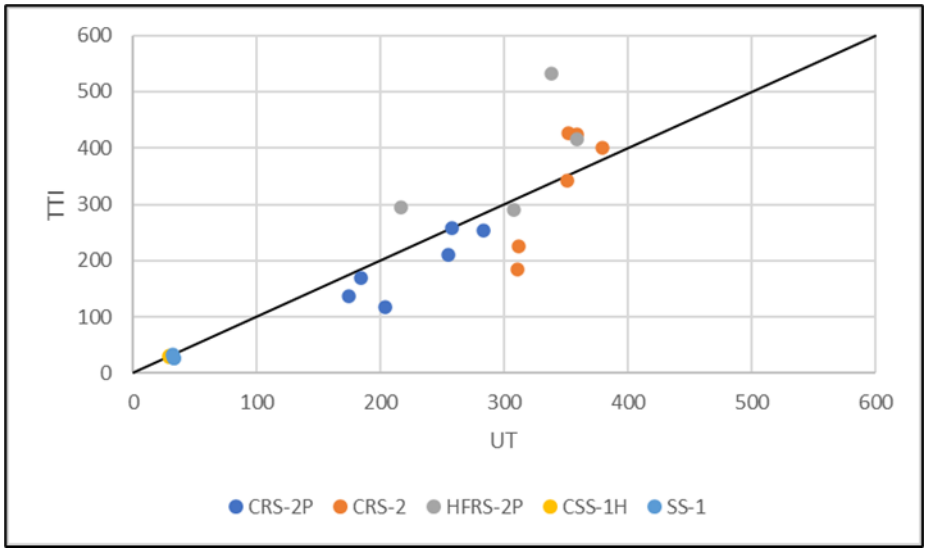


Figure 4.55. Comparison of rotational viscosity between the two labs.

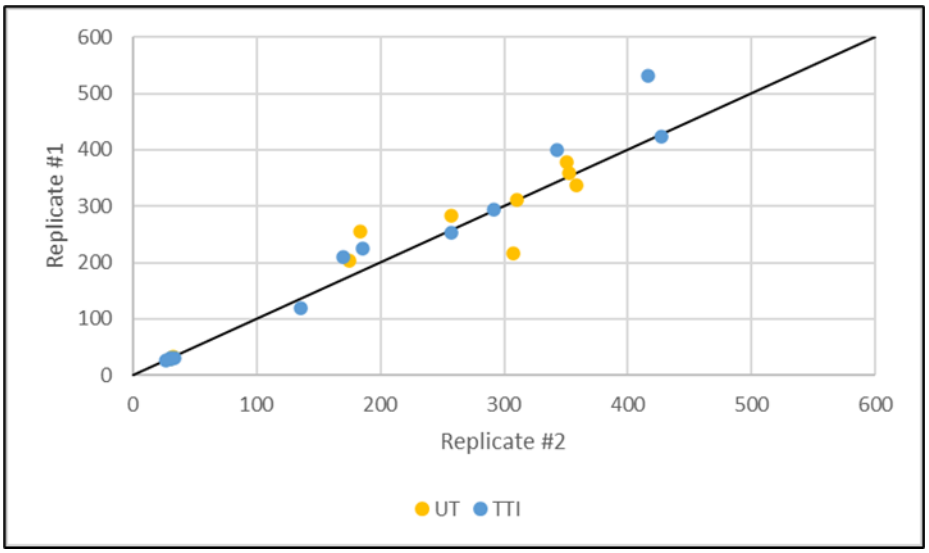


Figure 4.56. Comparison of rotational viscosity between two replicates.

4.5. Delta Tc

4.5.1. Goal

Current specification for evaluating the cracking behavior of asphalt binders requires stiffness and m-value testing at multiple (≥3) temperatures. The main goal of this exercise is to minimize

the propagation of errors arising from the compound variability from multiple measurements in the current spec by triggering a minimum stiffness requirement of S> 155 MPa when m-value falls below 0.32.

4.5.2. Scope

Currently, a requirement exists as a Special Specification in TxDOT SS3070 SuperPave Mixtures-Balanced Mix Design - PG Binders (Table 17).

4.5.3. Alternative Method

Figure 4.57 presents data from previous research on the feasibility of using the minimum BBR stiffness (S) requirement approach as a surrogate to ΔTc parameter. ΔTc and stiffness values were measured for a variety of asphalt binders from in and around the state of Texas. The proposed criterion is to require a minimum value of S (e.g. S > 155 MPa) when the value of m falls below a threshold (e.g. m < 0.32).

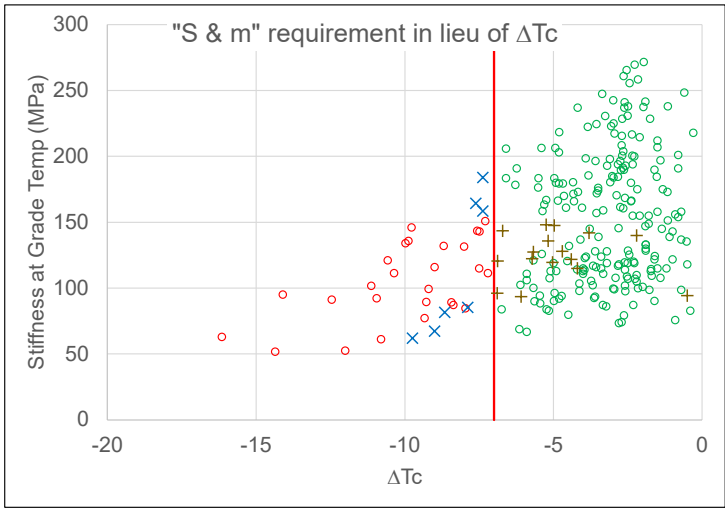


Figure 4.57 Stiffness versus ΔTc.

4.5.4. Conclusion

Based on current preliminary data from the BBR measurements, a new specification limit has been already proposed.

4.6. 8mm DSR for low temperature testing of PG binders

4.6.1. Goal

The main goal of this exercise is to track the use of G* and δ at low temperatures measured using an 8 mm DSR as a potential alternative to BBR S and m values.

4.6.2. Scope

No current requirement exists on this method using the DSR; however, the test is intended to serve as a surrogate for BBR.

4.6.3. Evaluation

4.6.3.1. Method and Test

The steps used for the low temperature testing of PG binders using the DSR are summarized below.

- The DSR temperature chamber was preheated to 70°C.
- The asphalt binder sample was transferred to one of the test plates, and the sample was tested at intermediate temperature according to AASHTO T315.
- After completion of 8 mm intermediate temperature testing, and without removing the sample, the temperature from the intermediate temperature was lowered to the low PG temperature plus 10°C, and the axial force adjustment of 0.3N (± 0.1N) in compression was used.
- The sample was conditioned at the test temperature (low PG grade temperature plus 10°C) for 2100 s.
- The specimen was tested at 0.1% strain rate and 0.2 rad/s angular frequency for ten cycles.
- The average G* and δ from the last five cycles at the PG low temperature grade plus 10°C was recorded.

It can be observed from the above procedure that this DSR procedure does not require an extra asphalt binder sample for low temperature testing. In fact, DSR procedure for intermediate temperature can be modified and programmed to add low temperature testing step to execute the low temperature testing on the same asphalt binder sample.

4.6.3.2. Materials

A variety of PG binders from different suppliers were used for the testing. They were as follows:

- Eight PG 64-22 binders
- Four PG 70-22 binders
- Seven PG 76-22 binders
- Two PG 58-28 binders
- Two PG 64-28 binders
- Four PG 70-28 binders
- One PG 76-28 binder

Three research labs participated in the testing: UT, TTI, and TxDOT. All the above-mentioned binders were tested in one of the research labs. Four asphalt binders were tested in all three labs: two PG 64-22 binders, one PG 70-22 binder, and one PG 76-22 binder. In most cases, two replicates were tested, but, in some cases, only replicate was tested.

4.6.4. Results and Discussion

Replicate data was not averaged and is shown as-is in the following figures. Figure 4.58 shows the G* values from DSR low temperature testing against stiffness values from BBR testing. The plot shows a good correlation between the stiffness values and G* values. Based on this correlation and using the BBR specification limit of 300 MPa a specification limit for G* can be obtained which corresponds to about 250 MPa. Quadrants were created in this plot based on these limits. Green quadrant (G*<250 MPa and BBR stiffness <300 MPa) corresponds to binders that pass both BBR stiffness test and DSR G* test. Red quadrant (G*>250 MPa and BBR stiffness >300 MPa) corresponds to binders failing both BBR stiffness test and DSR G* test. The remaining space corresponds to binders failing one of the two tests. Following observations can be made from this plot:

- All the binders tested fall into green quadrant. This shows that if the binders are passing the BBR stiffness test, they are also passing the DSR G* test, which means that DSR G* can be used as a parameter for low temperature performance testing.
- A more conservative limit of 220 MPa can be proposed where no BBR testing is required for DSR G* values less than 220 MPa for routine low temperature testing.
- BBR testing might only be required for binders with G* values greater than 220 MPa, as they are borderline cases which might lead to false positives or negatives.

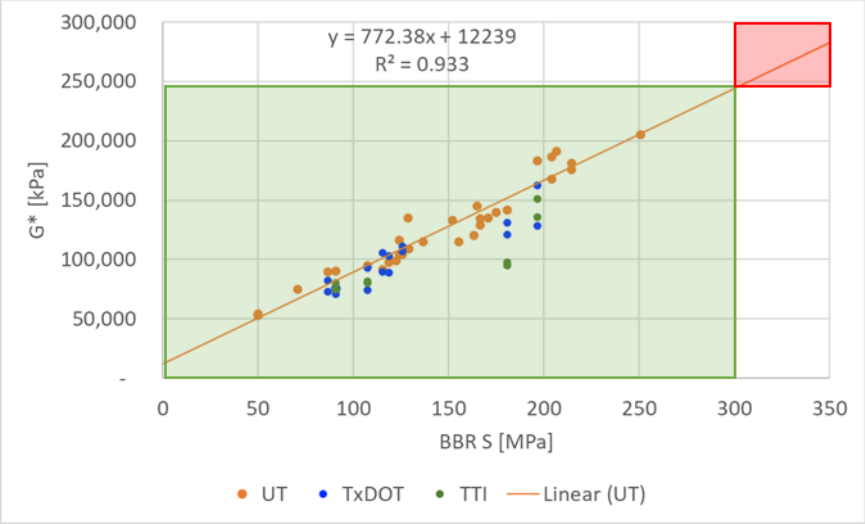


Figure 4.58. A plot showing G from DSR low temperature testing against the stiffness values from BBR testing.*

Figure 4.59 shows the phase angle (δ) value with BBR m-value. It can be observed that there is a good correlation between phase angle and BBR m-value. Using the specification limit for m-value of 0.3 and the correlation between phase angle and BBR-value, a specification limit on phase angle can be obtained as 22 degrees. Similar to the BBR stiffness and DSR G* plot, all binders falling in green quadrant (δ > 22 degrees and m-value > 0.3) pass both DSR phase angle and BBR m-value tests. All binders falling in red quadrant fail both tests (δ < 22 degrees and m-value < 0.3). Binders falling in the rest of the space fall one of the two tests. Based on the plot, following observations can be made:

- With the exception of one asphalt binder, all binders fall into the green quadrant, showing that if the binders are passing the BBR m-value test, they are also passing the DSR phase test. This means that DSR phase angle can be used as a parameter in the place of BBR m-value for low temperature performance testing.
- One asphalt binder that did not into the green quadrant, passed the DSR phase angle test, but failed the BBR m-value test in all the three labs (the points correspond to the same binder tested in three labs). An important note here is that this binder was very close to the pass/fail boundary.
- A conservative limit of phase angle > 22 degrees can be proposed for no BBR m-value testing to avoid false positives and negatives.
- BBR testing might be needed when the phase angle specification limit is not met.

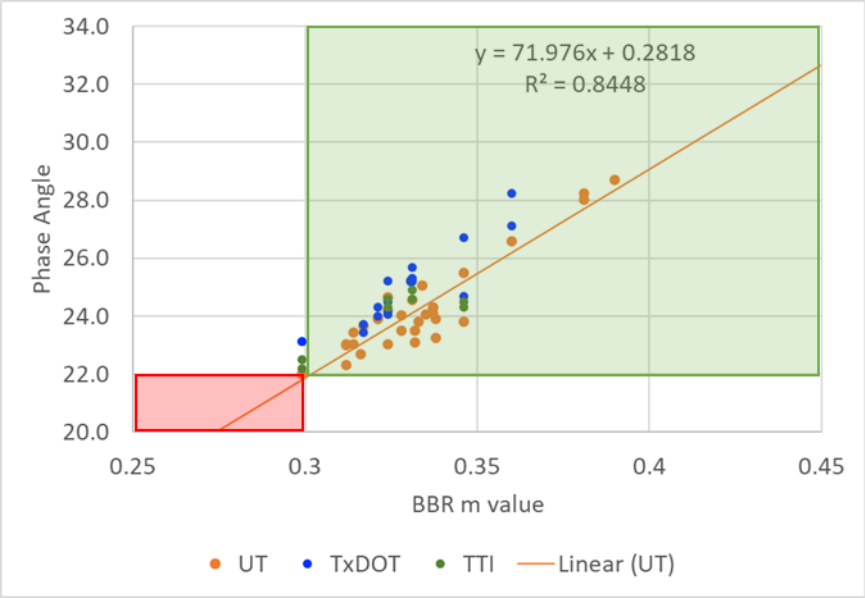


Figure 4.59. A plot showing phase from DSR low temperature testing against the m-value from BBR testing.

Figure 4.60 and Figure 4.61 show the plot of G* and phase angle of one replicate against the second replicate, respectively. It can be observed in both the plots that the data is close to the line of equality (1:1) showing good repeatability between the replicates both in G* and phase angle at low temperatures.

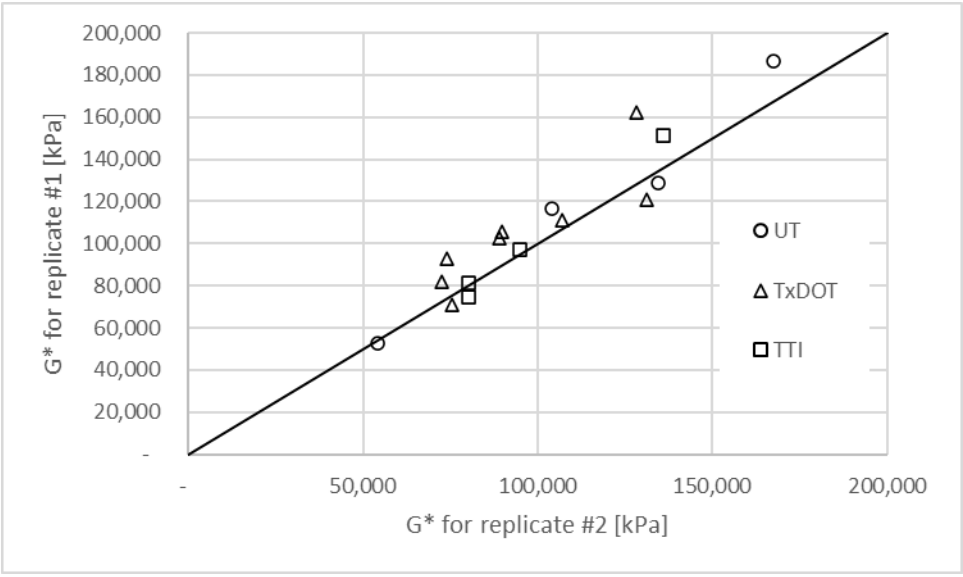


Figure 4.60. A plot showing G values obtained from two replicates (solid line is 1:1 line for reference).*

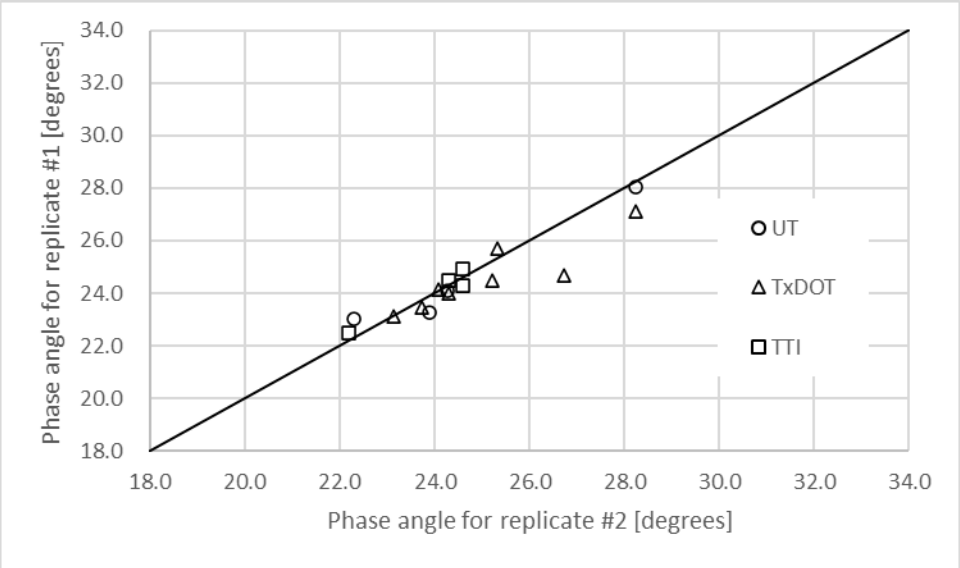


Figure 4.61. A plot showing phase angle values obtained from two replicates (solid line is 1:1 line for reference).

4.6.5. Low Temperature DSR Round Robin

A round robin was conducted with ten laboratories participating and eight different binder samples tested. Some labs conducted two separate tests, while some conducted only one test, resulting in sixteen data points for each binder (one sample was missing the results from two labs resulting in only 14 data points for that sample). The binders were chosen so that some met the PG grade easily, while others were borderline failing. The PG 76-28 was mislabeled and was actually a PG 76-22, but this represented testing a binder beyond its limits and it should have shown as not meeting the suggested specification limits.

All G* round robin datapoints for each binder are shown in Table 4.8.

Table 4.8. G* Round Robin Data.

G*(kPa)																		
Binder	PG grading	T(°C)	R1	R2	R3	R4	R5	R6	R7	R8	R9	R10	R11	R12	R13	R14	R15	R16
2467	PG64-22	-12	129989	134207	137380	126164		144882	143676	97380	127300	134971		74000	102656	93715	121463	135110
2930	PG70-22	-12	80575	78171	82239	64029	76575	93982	89964	84670	76950	91626	87326	38600	65808	56681	71745	84942
2939	PG76-22	-12	119531	111759	114909	115468	119091	138666	130064	107400	115900	133719	121260	56700	81790	80017	94519	124510
1017(2605)	PG76-22	-12	117286	129050	123852	93814	106375	131859	130733	128350	124470	125171	114202	65800	98469	72013	115512	126790
1423	PG64-28	-18	220136	214070	232319	198535	209742	252060	247517	212000	211800	224976	235167	86700	118466	148694	206917	231640
3467	PG64-28	-18	185729	164656	187220	161253	101660	184796	181967	160200	121600	183172	193160	45000	103456	104882	122550	189950
2534	PG70-28	-18	131682	158830	156314	158713	147176	173026	178377	155400	137800	153871	143105	60600	85219	90466	122146	159090
0970	PG76-28	-18	211621	225956	243578	174368	236140	243705	250221	192900	211500	223257	216017	111000	132506	138057	199980	239200

Figure 4.62 shows all test results for all samples for G*. A suggested specification limit of 220,000 kPa line is also shown. This would be a suggested maximum specification limit.

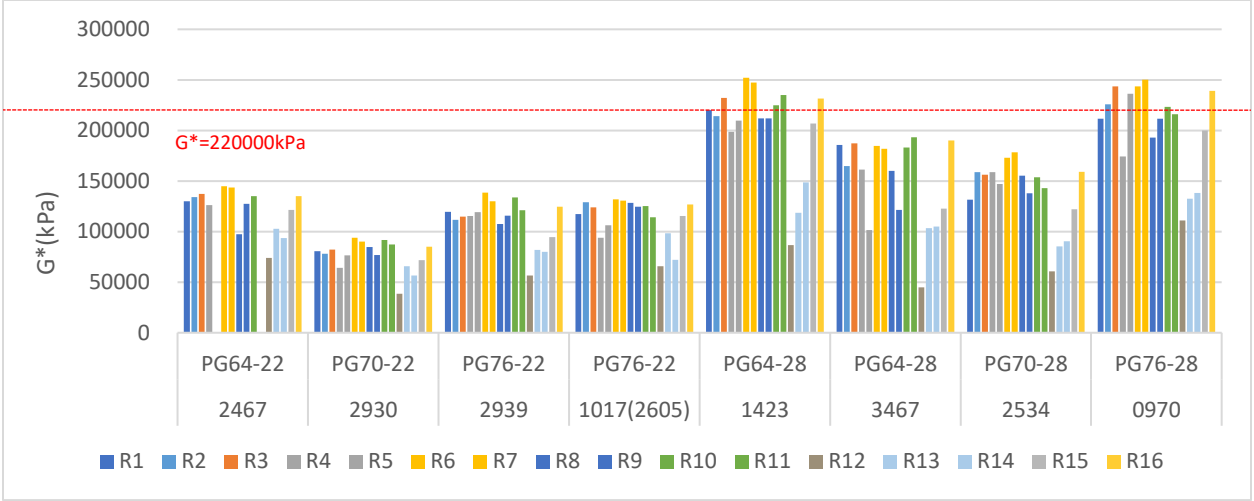


Figure 4.62. Round Robin G for all samples.*

All Phase Angle (δ) round robin datapoints for each binder are shown in Table 4.9.

Table 4.9. Phase Angle (δ) Round Robin Data.

δ(°)																		
Binder	PG grading	T(°C)	R1	R2	R3	R4	R5	R6	R7	R8	R9	R10	R11	R12	R13	R14	R15	R16
2467	PG64-22	-12	26.1	25.8	25.2	25.2		24.8	24.8	25.7	25.8	25.5		30.7	27.0	27.2	26.0	24.8
2930	PG70-22	-12	22.8	23.2	22.4	22.6	22.5	22.5	22.5	22.9	22.9	22.7	22.7	25.8	23.5	24.2	23.1	22.2
2939	PG76-22	-12	25.4	25.7	25.0	25.0	25.0	24.6	24.7	25.6	25.6	25.0	24.9	31.3	27.3	27.6	27.9	24.4
1017(2605)	PG76-22	-12	22.5	22.1	22.2	22.3	22.3	22.1	22.1	22.5	22.6	22.3	22.4	26.6	23.2	23.1	22.4	21.8
1423	PG64-28	-18	22.2	22.0	20.9	21.6	21.9	20.8	20.8	22.1	22.3	21.3	21.2	31.0	26.7	25.9	22.1	20.6
3467	PG64-28	-18	26.7	28.8	27.0	25.2	25.1	24.7	24.8	27.3	25.5	25.2	25.3	41.1	32.4	34.4	27.9	24.2
2534	PG70-28	-18	25.4	24.3	23.7	24.0	24.3	23.5	23.4	24.8	24.9	23.8	23.9	31.5	28.5	28.6	24.8	23.7
0970	PG76-28	-18	19.2	18.4	18.0	18.8	18.7	18.1	18.2	19.0	19.0	18.6	18.4	23.7	21.1	21.1	18.8	17.9

Figure 4.63 shows all test results for all samples for phase angle. A suggested specification limit of 22 degrees is also shown. This would be a suggested minimum specification limit.

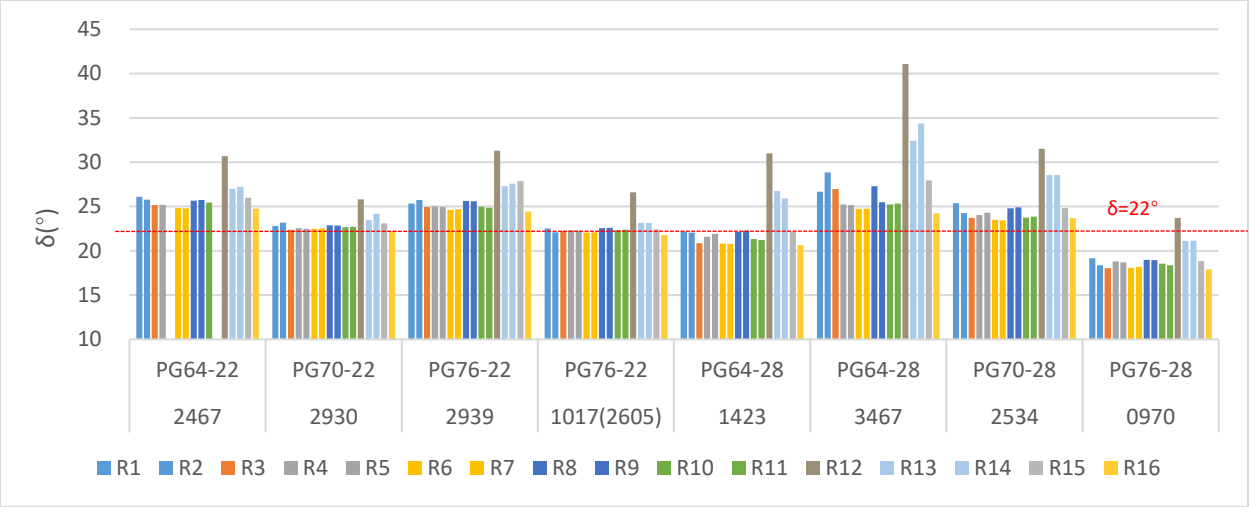


Figure 4.63. Round Robin Phase Angle for all samples.

The G* was met for some labs and failed for others. The δ did not meet the suggested limits across the board, except for one lab showing a higher δ. This was by design according to the materials selected for the round robin.

One can see that there are some data points, both high and low and for G* and δ, that appear to be outliers. There were some labs that were consistently high or low. These may have had systematic issues, such as temperature calibration. Results were analyzed to calculate averages, standard deviations, z-scores (how many standard deviations away from the average), and coefficients of variation (standard deviation divided by the mean). Data points that appeared to be outliers because of z-scores were removed, and the statistics recalculated. This information is shown in Table 4.10.

Table 4.10. G* and Phase Angle (δ) Statistics With and Without Outliers

Statistics With and Without Outliers								
Binder	2467	2930	2939	1017(2605	1423	3467	2534	0970
PG grading	PG64-22	PG70-22	PG76-22	PG76-22	PG64-28	PG64-28	PG70-28	PG76-28
T(°C)	-12	-12	-12	-12	-18	-18	-18	-18
G*(kPa) Statistics All Data								
Mean	121635	76493	110331	112734	203171	149453	138239	203125
StDev	21324	14480	21863	20505	46088	43983	33158	43032
COV	17.5%	18.9%	19.8%	18.2%	22.7%	29.4%	24.0%	21.2%
G*(kPa) Statistics Outliers Removed								
Mean	133514	83230	118984	122804	222837	179210	154449	224506
StDev	7475	6759	11499	7790	16140	12323	13343	18479
COV	5.6%	8.1%	9.7%	6.3%	7.2%	6.9%	8.6%	8.2%
δ(°) Statistics (All Data)								
Mean	26.03	23.02	25.93	23.11	22.72	27.86	25.19	19.18
StDev	1.54	0.89	1.79	1.62	2.80	4.53	2.31	1.54
COV	5.9%	3.9%	6.9%	7.0%	12.3%	16.3%	9.2%	8.0%
δ(°) Statistics (Outliers Removed)								
Mean	25.41	22.83	25.07	22.27	21.53	25.99	24.19	18.53
StDev	0.48	0.50	0.42	0.23	0.61	1.41	0.61	0.41
COV	1.9%	2.2%	1.7%	1.0%	2.8%	5.4%	2.5%	2.2%

This analysis was the basis for removing the outlier data as shown in Table 4.11 and replotting the remaining data for G* and δ in Figure 4.64 and Figure 4.65. Removing the outliers reduced the COV for G* by a factor of 2 to 4 and the COV for Phase Angle (δ) by a factor of 2 to 7.

Table 4.11. Round Robin Data with Outliers Removed.

nder	PG grading	T(°C)	R1	R2	R3	R4	R5	R6	R7	R8	R9	R10	R11	R12	R13	R14	R15	R16
G*(kPa)																		
2467	PG64-22	-12	129989	134207	137380	126164		144882	143676		127300	134971					121463	135110
2930	PG70-22	-12	80575	78171	82239		76575	93982	89964	84670	76950	91626	87326				71745	84942
2939	PG76-22	-12	119531	111759	114909	115468	119091	138666	130064	107400	115900	133719	121260				94519	124510
1017(2605)	PG76-22	-12	117286	129050	123852		106375	131859	130733	128350	124470	125171	114202				115512	126790
1423	PG64-28	-18	220136	214070	232319	198535	209742	252060	247517	212000	211800	224976	235167				206917	231640
3467	PG64-28	-18	185729	164656	187220	161253		184796	181967	160200		183172	193160					189950
2534	PG70-28	-18	131682	158830	156314	158713	147176	173026	178377	155400	137800	153871	143105					159090
0970	PG76-28	-18	211621	225956	243578		236140	243705	250221	192900	211500	223257	216017				199980	239200
δ(°)																		
2467	PG64-22	-12	26.1	25.8	25.2	25.2		24.8	24.8	25.7	25.8	25.5					26.0	24.8
2930	PG70-22	-12	22.8	23.2	22.4	22.6	22.5	22.5	22.5	22.9	22.9	22.7	22.7				23.1	22.2
2939	PG76-22	-12	25.4	25.7	25.0	25.0	25.0	24.6	24.7	25.6	25.6	25.0	24.9					24.4
1017(2605)	PG76-22	-12	22.5	22.1	22.2	22.3	22.3	22.1	22.1	22.5	22.6	22.3	22.4				22.4	21.8
1423	PG64-28	-18	22.2	22.0	20.9	21.6	21.9	20.8	20.8	22.1	22.3	21.3	21.2				22.1	20.6
3467	PG64-28	-18	26.7	28.8	27.0	25.2	25.1	24.7	24.8	27.3	25.5	25.2	25.3				27.9	24.2
2534	PG70-28	-18	25.4	24.3	23.7	24.0	24.3	23.5	23.4	24.8	24.9	23.8	23.9				24.818	23.7
0970	PG76-28	-18	19.2	18.4	18.0	18.8	18.7	18.1	18.2	19.0	19.0	18.6	18.4				18.8	17.9

No Data Supplied
Removed as Outlier

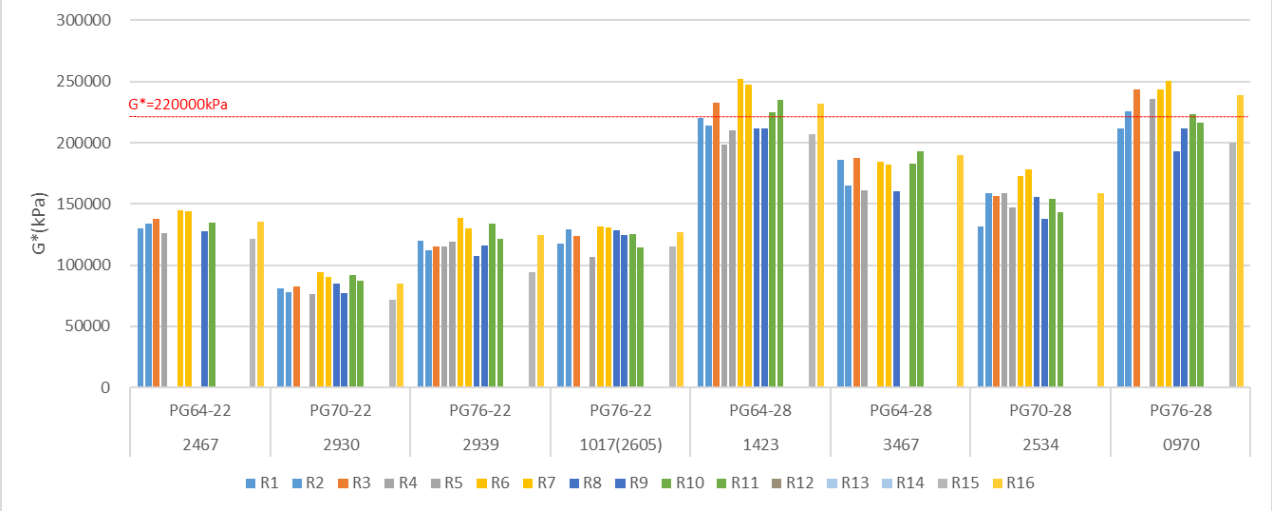


Figure 4.64. Round Robin G Data with Outliers Removed with Suggested Specification Limit.*

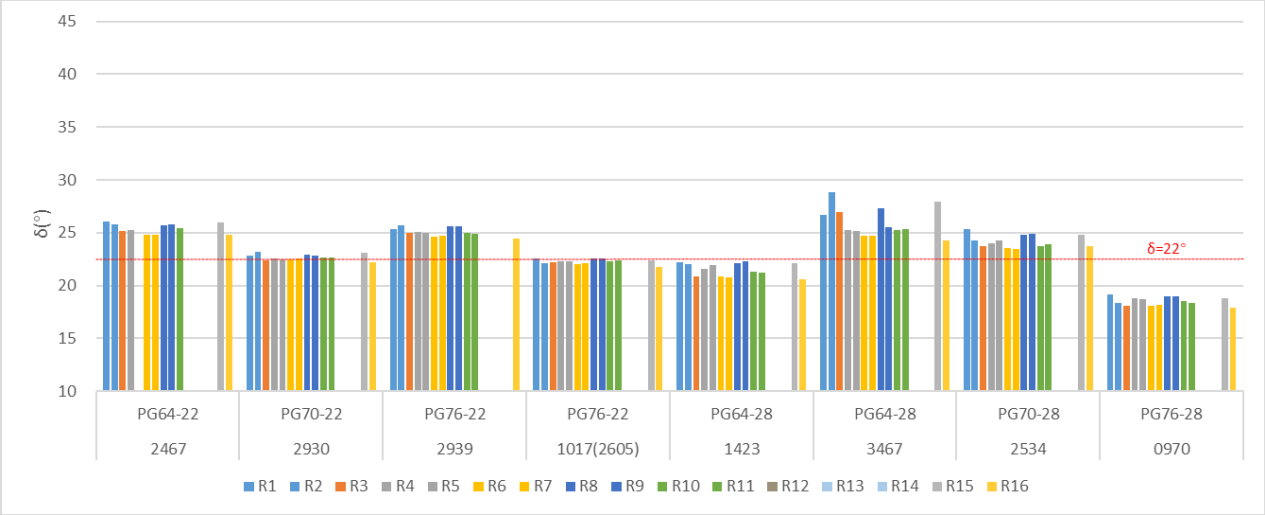


Figure 4.65. Round Robin Phase Angle (d) Data with Outliers Removed with Suggested Specification Limit.

The data show good repeatability with low COVs when the few labs showing as outliers are eliminated. It is believed that the outlier problem is mainly due to temperature calibration issues and an unfamiliar test procedure. Results are expected to improve with experience in performing the test.

4.6.6. Conclusion

Overall, a good correlation of G* and phase angle was observed with BBR stiffness and m-value, respectively. A good repeatability between the labs was observed. From the data collected, specification limits of G*< 220 MPa and δ > 22 degrees are suggested for routine screening of PG binders for low temperature testing in place of BBR. BBR testing is required in case the binders do not meet these specification limits.

4.7. Poker-chip test for intermediate temperature testing of PG binders

4.7.1. Goal

The main goal of this exercise is to track the poker chip strength and ductility parameter at intermediate temperature in order to use them as a potential alternative to the G*sin(δ) fatigue parameter at intermediate temperature.

4.7.2. Scope

No current requirement exists on this method, but it is intended to replace ductility requirements and surrogate ductility requirements, such as elastic recovery.

4.7.3. Evaluation

4.7.3.1. Method and Test

The poker-chip test sample preparation steps are summarized below:

1. Asphalt binder sample is heated in an oven to 320 ± 5°F (160 ± 3°C) for 15 to 20 minutes until the binder is fluid enough to be poured.
2. The binder sample is stirred thoroughly to ensure it is homogenous. 4.5 ± 0.05 g of the asphalt binder is poured inside the silicon mold as shown in Figure 4.66, and it is then allowed to cool at room temperature for about 20 minutes.

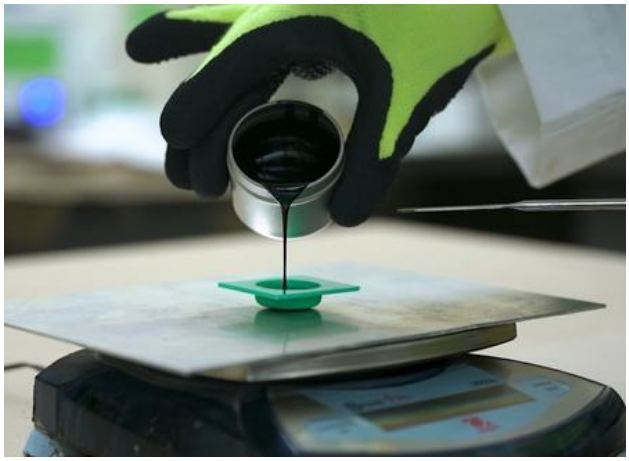


Figure 4.66. Asphalt binder poured inside the silicone mold.

3. The top and bottom parts of the poker chip mold and the spacer dowels are preheated in a conventional oven at 160 ± 3°C for at least three hours.

4. The pre-weighed dollop of the asphalt binder is then removed from the silicon mold and placed in the bottom part of the poker chip mold (Figure 4.67). The bottom part of the mold is then placed back in the oven at 160 ± 3°C for 15 minutes or until the dollop of asphalt binder is completely molten and spread over the entire bottom surface of the poker chip mold.

Figure 4.67. Bottom plates for five poker-chip samples (left) and one bottom plate with a binder sample placed in it (right).

5. Three spacer dowels are placed approximately at 120 degrees apart and about halfway radially out from the center of the bottom poker-chip sample disc (Figure 4.68).

Figure 4.68. Bottom plates for five poker-chip sample discs with binder samples (left) and one bottom plate with the three spacer dowels dropped on top of the binder surface (right).

6. The bottom part of the poker chip mold is placed back in the oven again for another 15 to 20 minutes to allow the spacer dowels to sink and submerge in the binder.

7. The bottom part and the pre-heated top part of the molds are taken out from the oven. Two alignment pins are then inserted in the bottom part of the mold. The top part of the poker chip mold is slowly lowered through the alignment pins and firmly pressed at the center. The third alignment pin is inserted to ensure proper alignment (Figure 4.69).

Figure 4.69. A poker-chip sample that sandwiches the binder specimen between the top and bottom pieces that are aligned using three pins (left); five complete discs ready for temperature conditioning and testing (right).

8. The poker-chip assembly is then placed in the oven at 160 ± 3°C for another 20 to 25 minutes.

9. The poker-chip assembly is taken out of the oven and allowed to cool to room temperature on a level platform for at least 30 min.

10. The poker-chip assembly is placed inside a temperature-controlled chamber at 25 ± 0.5°C until the specimen has reached the temperature. The poker-chip sample is then tested within 48 hours of conditioning.

Poker-chip sample testing is summarized in the step below:

1. The load frame is programmed to run the following sequence: i) Apply tension in displacement-controlled mode of 1 mm/minute until a tensile load of 40 N is reached. At this point, ii) switch to load-controlled mode and apply a tensile load of 2 N/second until the sample fails.

2. The alignment pins from the specimen are gently removed while holding the bottom of the specimen. Then, the bottom of the poker chip assembly is locked in the loading frame.

3. The loading axis is gently lowered until the clevis rod end hole aligns with the hole in the ball joint rod. A dowel is gently inserted through the clevis rod end and the ball joint rod, locking the two in place. Once the sample is locked in place, the programmed test is executed.

4. The load and displacement at a rate of at least two points per second are recorded during the test.

5. The two parameters of interest that are extracted and evaluated in this study are (i) Strength parameter: tensile strength of the binder, and (ii) Ductility parameter: elongation or percentage strain of the binder when it reaches 80% of post peak stress (Figure 4.70).

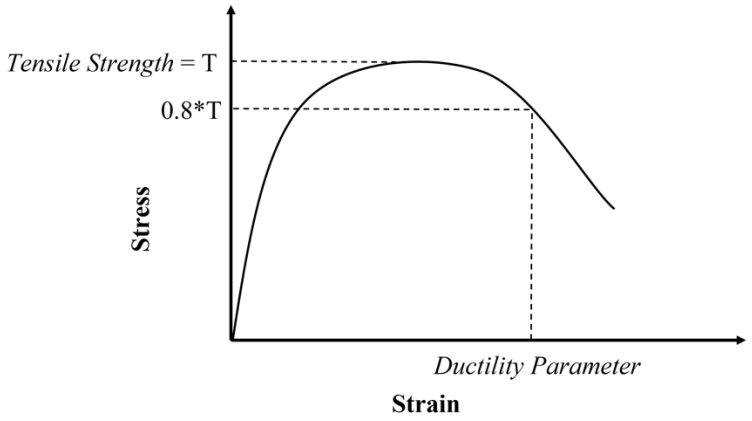


Figure 4.70. Typical stress-strain curve with the two parameters of interest (i) tensile strength and (ii) ductility.

4.7.3.2. Materials

Twelve asphalt binders were used for round robin testing in three research laboratories: UT, TTI and TxDOT. The asphalt binders were of the following grade:

- PG 64-22 from two different sources
- PG 70-22 from two different sources
- PG 76-22 from two different sources
- PG 64-28 from two different sources
- PG 70-28 from three different sources
- PG 58-28 from one source

4.7.4. Results and Discussion

The strength and ductility parameters obtained from the poker-chip test on RTFO aged binders were compared from the three participating labs. Please note that the round-robin testing was conducted at room temperature in the TTI and TxDOT labs. In case of the UT lab, the samples were conditioned overnight at 25°C and tested at room temperature.

4.7.4.1. Strength and ductility parameter

Figure 4.71 to Figure 4.73 show the comparison of poker-chip test strength parameter between the three participating labs. It can be observed in all three figures that the data is close to the line of equality (1:1), showing an overall good repeatability of strength parameter between the participating laboratories. Figure 4.74 to Figure 4.76 show the comparison of poker-chip ductility parameter between the participating labs. Similar to poker-chip strength data, poker-chip ductility data is also close to the line of equality, showing an overall good repeatability between the three labs.

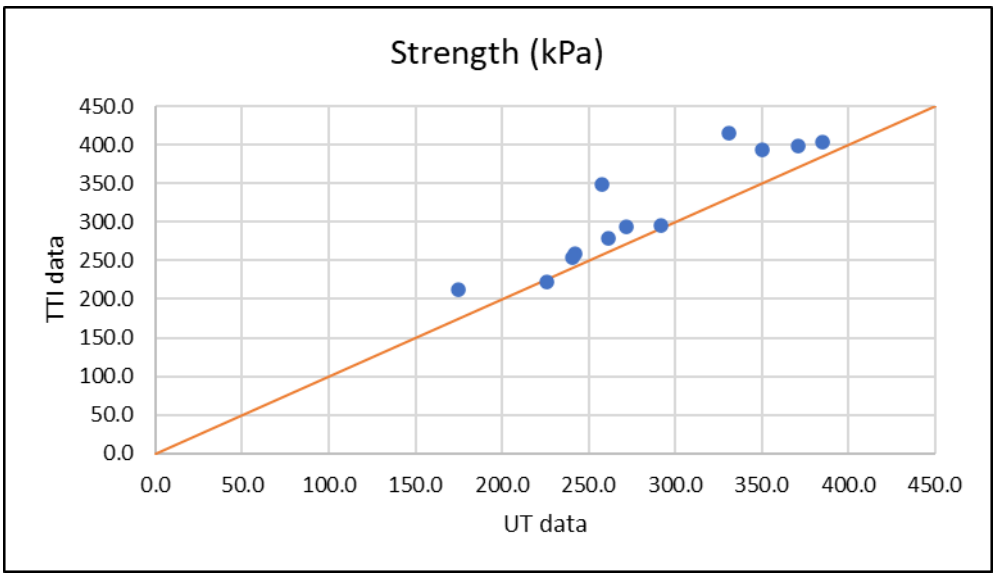


Figure 4.71. Comparison of strength parameter from poker-chip test between UT lab and TTI lab (Orange line shows the 1:1 line for reference).

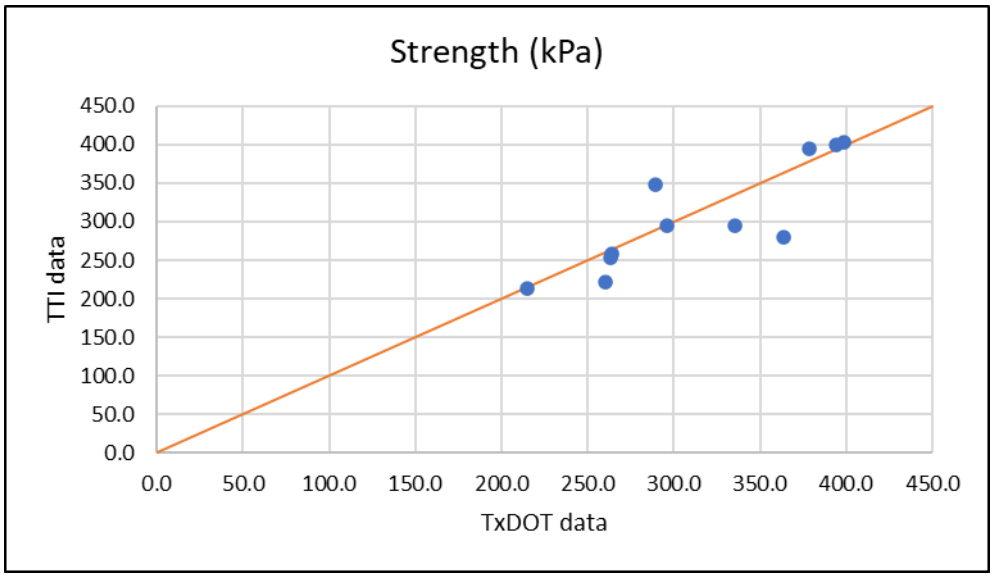


Figure 4.72. Comparison of strength parameter from poker-chip test between TxDOT lab and TTI lab (Orange line shows the 1:1 line for reference).

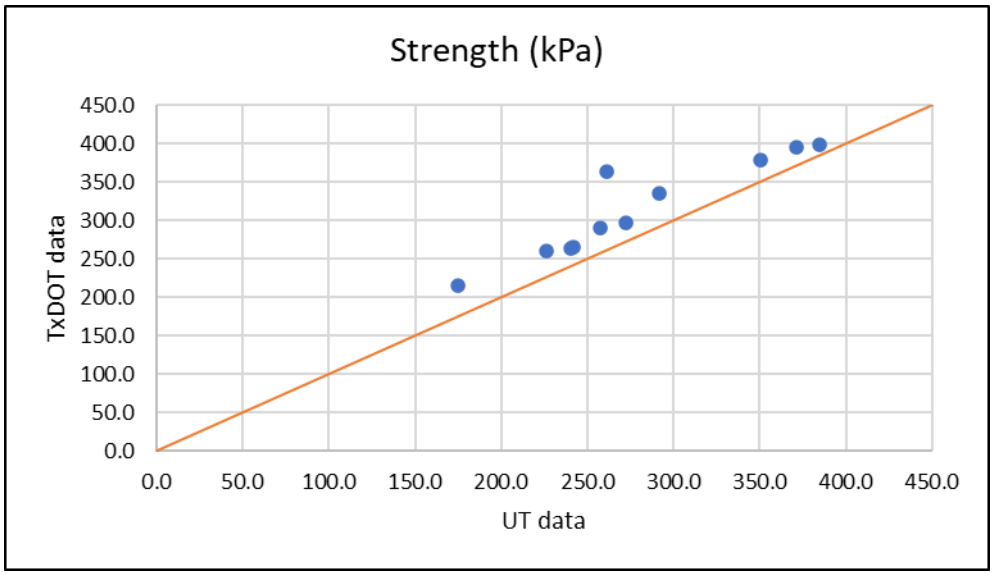


Figure 4.73. Comparison of strength parameter from poker-chip test between TxDOT lab and UT lab (Orange line shows the 1:1 line for reference).

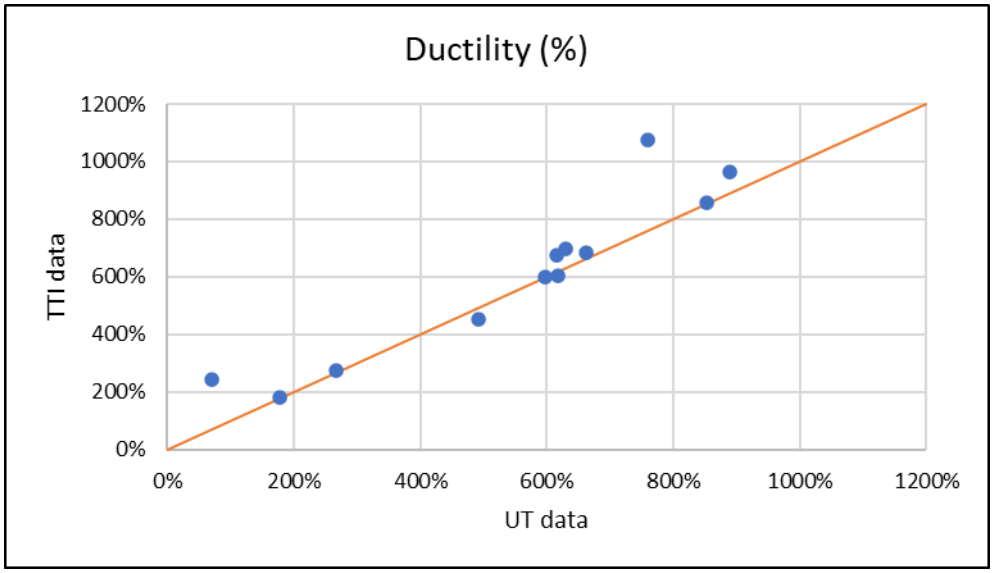


Figure 4.74. Comparison of ductility parameter from poker-chip test between UT lab and TTI lab (Orange line shows the 1:1 line for reference).

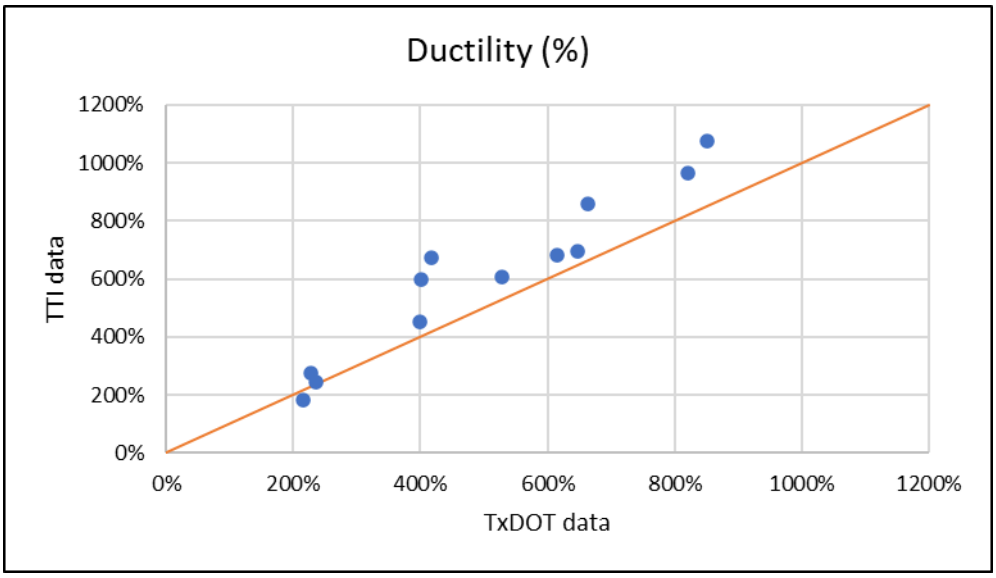


Figure 4.75. Comparison of ductility parameter from poker-chip test between TxDOT lab and TTI (Orange line shows the 1:1 line for reference).

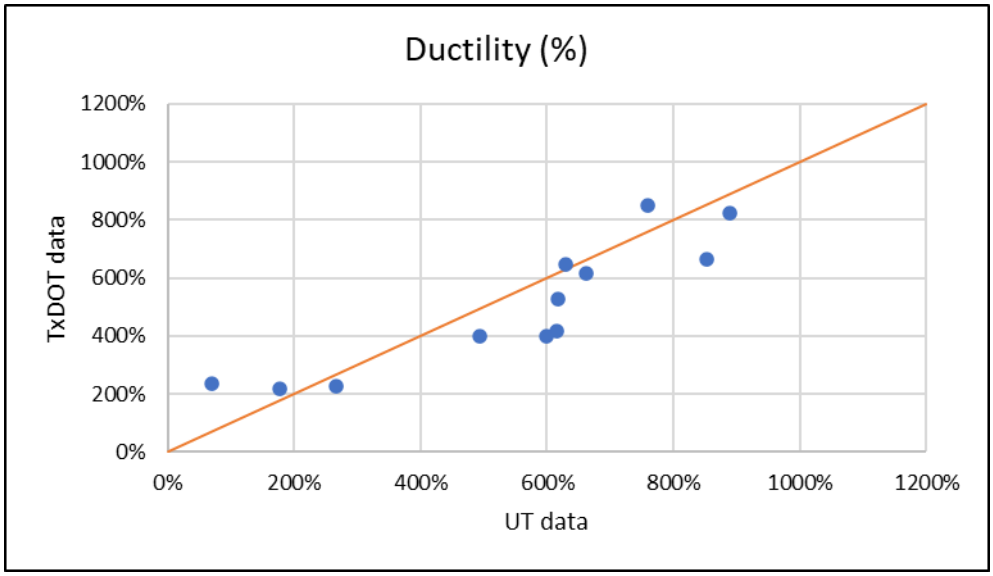


Figure 4.76. Comparison of ductility parameter from poker-chip test between TxDOT lab and UT (Orange line shows the 1:1 line for reference).

4.7.4.2. Variability in strength and ductility parameters

4.7.4.2.1. Variability between replicates

The d2s% (absolute value of the difference of two values x 100/average of the two values) was calculated for the replicates in all three laboratories for poker-chip strength and ductility parameters. The following observations can be made from the d2s% values:

- The variability in ductility parameter is more as compared to the strength parameter.

- The d2s% within replicates for strength parameter is within 30% with one exception.
- The d2s% within replicates for ductility is typically below 20% with a few exceptions. Usually, a higher variability in ductility is observed in modified asphalt binders due to very high ductility values.

4.7.4.2.2. Variability between labs

The inter-laboratory variability in poker-chip strength and ductility parameter was also evaluated. Minimum and maximum d2s% were calculated using all the combinations of inter-laboratory tests. This allowed for observation of the range of d2s% possible for tests between labs. The following observations can be made from the d2s% values:

- The inter-laboratory variability for strength parameter is less than the ductility parameter, similar to within-replicates variability.
- The minimum variability for both strength and ductility parameters is close to zero.
- The highest d2s% observed for strength parameter is around 60%, but in most cases, it was below 30%.
- The highest d2s% observed for ductility parameter is around 140%, but in most cases, it was below 30%. A high variability in ductility parameter is generally observed in the case of modified binders due to very high ductility values (with one exception).

4.7.4.3. Effect of temperature on poker-chip test

The poker-chip test was conducted at different temperatures to observe the effect of temperature on the strength and ductility parameter. This testing was conducted at the UT lab only. For this testing, the samples were conditioned for the corresponding temperature overnight and tested at the same temperature. PG 64-28 binder was used for this testing. The testing was conducted with two replicates at five different temperatures: 20°C, 23°C, 25°C, 27°C, and 30°C. Figure 4.77 and Figure 4.78 show the strength and ductility parameter, respectively. The following observations were made from these figures:

- A clear pattern of strength parameter going down with the increase in temperature can be observed from Figure 4.77.
- No pattern was observed for ductility parameter with temperature. Due to very high values in ductility of the modified binder used in the testing, the pattern in ductility parameter is not observed.
- The variability in ductility parameter within replicates is high due to very high values of ductility in the modified asphalt binder.

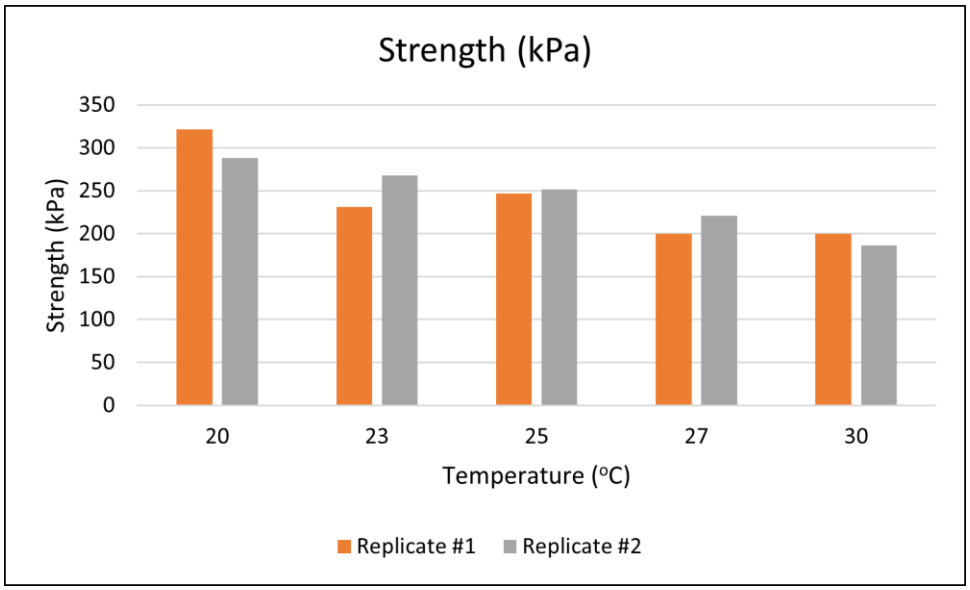


Figure 4.77. Effect of temperature on poker-chip test strength parameter.

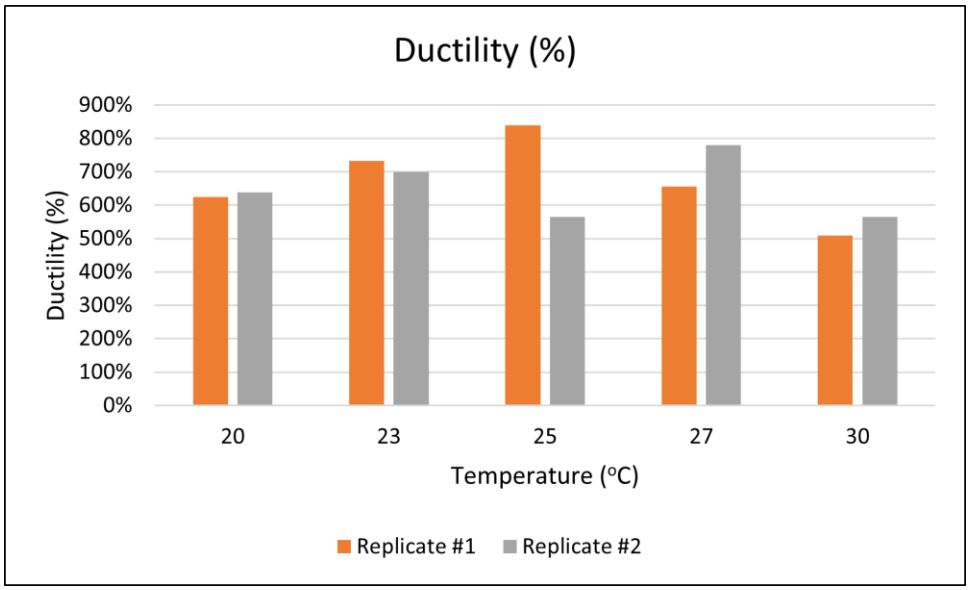


Figure 4.78. Effect of temperature on poker-chip ductility parameter.

4.7.5. Conclusions and Recommendations

Overall, a good repeatability was observed between the three participating labs. The repeatability between two replicates was also good. The effect of temperature on strength parameter is more significant compared to the ductility parameter. Suggested ductility parameter specification limits for RTFO-aged binders are:

- 70-22 and 76-22 = 400%
- 64-28 and 70-28 = 600%

- 76-28 = 800%

Chapter 5. Draft of Revised Item 300

The recommended specification limits in the following sections are based on limited data or extrapolations. In particular, DSR values for cut back and emulsion residues need to be further evaluated, and values for Saybolt viscosity do, as well. Additionally, the low temperature DSR test has been codified in Tex-554-C, and vacuum oven recovery for emulsions and cutbacks has been codified in Tex-555-C.

The suggested table-by-table changes in the Item 300 specifications are described in the sections below.

5.1. Table 2 Asphalt Cement

- Delete unused columns.
- For original and RTFOT aged binder:
- Replace Absolute Viscosity at 140°F and 275°F with Rotational Viscosity at 140°F and 275°F.
- Replace AASHTO T 202, “Standard Method of Test for Viscosity of Asphalts by Vacuum Capillary Viscometer” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.” This is both for original and RTFO aged binder.
- Additional data gathering is required to recommend specification thresholds for different grades.
- For original binder:
- Eliminate Penetration (given that these grades are used to produce cold patch mixes).

Table 2 of Item 300 after the proposed changes is below:

Table 2

Asphalt Cement

Property	Test Procedure	Viscosity Grade			
		AC-0.6		AC-1.5	
		Min	Max	Min	Max
Viscosity					
140°F, Pa·s	T 316	4.0	8.0	10.0	20.0
275°F, Pa·s		0.04	–	0.07	–
Flash point, C.O.C., °F	T 48	425	–	425	–
Solubility, %	T 44	99	–	99	–
Spot test	Tex-509-C	Neg.		Neg.	
Tests on residue from RTFOT:	T 240				
Viscosity, 140°F, Pa·s	T 316	–	30.0	–	100.0
Poker Chip, Ductility, % [1]	TP 150	200	–	200	–

1. Need to validate.

5.2. Table 3 Polymer-Modified Asphalt Cement

1. Delete Absolute Viscosity at 140°F.

- These materials, except AC-15P, all have a DSR requirement already. Absolute viscosity at 140°F is a redundant test.

- For AC-15P and AC-12-5TR, add a requirement for DSR of 1.0 at 58°C.

2. Delete Penetration altogether – PG binders do not have this requirement, only DSR at intermediate temperature on RTFO+PAV residue.

3. Replace Elastic Recovery with MSCR, T350, Recovery, 0.1 kPa, at DSR High Temperature, % Min.

- Replace Tex-539-C “Measurement of Elastic Recovery of Tensile Deformation using a Ductilometer” with AASHTO T350 “Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).” This would measure the recovery at 0.1kPa, as tested at the DSR test temperature.

- Additional data gathering is required to recommend specification limits. Tentatively, these may be the same as for PG binders.

4. Add 8mm, Low Temperature DSR as surrogate for BBR.

- Add Tex-55X-C, "Low Temperature DSR as a Surrogate for S and M-value" (Actual title to be determined). This would be 8mm DSR at PG Low + 10°C (in this case, -18°C) with phase angle > 22.0 degrees and G* < 220,000 kPa. Add a footnote that a binder must meet either low temp DSR requirements or BBR S and m-value.

Table 3 of Item 300 after proposed changes is below:

Table 3
Polymer-Modified Asphalt Cement

Property	Test Procedure	Polymer-Modified Viscosity Grade											
		AC-12-5TR		NT-HA[1]		AC-15P		AC-20XP		AC-10-2TR		AC-20-5TR	
		Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Polymer		TR		–		SBS		SBS		TR		TR	
Polymer content, % (solids basis)	Tex-533-C or Tex-553-C	5	–	–	–	3	–	–	–	2	–	5	–
Dynamic shear, G*/sin(d), Min, 1.00 kPa, Test temperature @ 10 rad/sec., °C	T 315	58		82		58		64		58		64	
MSCR Recovery, 0.1 kPa, DSR Temperature, % Min	T350	30				30	–	30	–	20	–	30	–
Viscosity, 275°F, Pa-s	T 316	–	0.8	–	4.0	–	0.8	–	0.8	–	0.8	–	1
Polymer separation, D,%	Tex-540-C	-10	+10	–	–	-10	+10	-10	+10	-10	+10	-10	+10
Flash point, C.O.C., °F	T 48	425		425		425	–	425	–	425	–	425	–
Tests on residue from RTFOT aging and pressure aging:	T 240 and R 28												
Creep stiffness [2]	T 313												
S, -18°C, MPa		–	300	–	–	–	300	–	300	–	300	–	300
m-value, -18°C		0.300	–	–	–	0.300	–	0.300	–	0.300	–	0.300	–
Low Temperature DSR, -18C [2]	Tex-554-C												
Phase angle (δ), degrees		22.0		–	–	22.0		22.0		22.0		22.0	
G*, MPa			220	–	–		220		220		220		220

1. This is a hot-applied TRAIL product.
2. If RTFO-PAV aged binder meets Low Temperature DSR, Creep Stiffness testing is not required.

5.3. Table 4 Rapid-Curing Cutback Asphalt (RC-250 is the only remaining RC Cutback)

1. Delete Columns not used.

2. Replace Kinematic Viscosity with Rotational Viscosity.

- Replace AASHTO T 201, "Standard Method of Test for Kinematic Viscosity of Asphalts (Bitumens)" with AASHTO T 316, "Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer." Additional data are being gathered to recommend a specification limit. The current suggestion is to leave the same limits. These are slightly different than cSt old units, but are related by the specific gravity. Producers may make slight modifications to materials to meet these limits.
- Additional data gathering is required to recommend specification limits.

3. Keep Distillation as reserve test and not for routine testing.

4. Add Vacuum Oven for standard testing of residue % and testing of residue.

- Add Tex-555-C, "Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven."

5. For Vacuum Recovery of Residue:

- Replace Absolute Viscosity with DSR.
- Replace AASHTO T 202, "Standard Method of Test for Viscosity of Asphalts by Vacuum Capillary Viscometer" with AASHTO T 315, "Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)."
- Conditions and criteria/thresholds for different grades need to be developed.

6. Delete Ductility.

- Delete AASHTO T51, "Standard Method of Test for Ductility of Asphalt Materials."

Table 4 of Item 300 after proposed changes is below:

Table 4

Rapid-Curing Cutback Asphalt

Property	Test Procedure	Type–Grade	
		RC-250	
		Min	Max
Viscosity, 60°C, cP	T 316	250	400
Water, %	D95	–	0.2
Flash point, T.O.C., °F	T 79	80	–
Distillation test: [1]	T 78		

Distillate, percentage by volume of total distillate to 680°F			
to 437°F		40	75
to 500°F		65	90
to 600°F		85	–
Residue from distillation, volume %		70	–
Vacuum Recovery Test	Tex-555-C		
Residue from Vacuum Recovery Test, volume %		70	
Tests on Vacuum Recovered residue:			
Dynamic shear, G*/sinδ, 58°C, 10 rad/s, kPa	T 315	0.50	3.00
Solubility, %	T 44	99	–
Spot test	Tex-509-C	Neg.	

1 The distillation test is reserved for testing as needed or forensic purposes.

5.4. Table 5 Medium-Curing Cutback Asphalt

1. Delete Columns not used.
2. Delete MC-800 and MC-3000 materials. This leaves MC-30 as the only remaining Medium Curing Cutback.

Additionally for MC-30:

3. Replace Kinematic Viscosity with Rotational Viscosity.

- Replace AASHTO T 201, “Standard Method of Test for Kinematic Viscosity of Asphalts (Bitumens)” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.” Additional data are being gathered to recommend a specification limit. Current suggestion is to leave the same limits. These are slightly different than cSt old units but are related by the specific gravity. Producers may make slight modifications to materials to meet these limits.
- Additional data gathering is required to recommend specification limits.

4. Keep Distillation as reserve test and not for routine testing.

- Add Vacuum Oven for standard testing of residue % and testing of residue.
- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

5. For Vacuum Recovery of Residue:

- Replace Absolute Viscosity with DSR.
- Replace AASHTO T 202, “Standard Method of Test for Viscosity of Asphalts by Vacuum Capillary Viscometer” with AASHTO T 315, “Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).”
- Additional data gathering is required to recommend specification limits.

6. Delete Ductility.

- Delete AASHTO T51, “Standard Method of Test for Ductility of Asphalt Materials.”

Table 5 of Item 300 after proposed changes:

Table 5

Medium-Curing Cutback Asphalt

Property	Test Procedure	Type–Grade MC-30 Min	Type–Grade MC-30 Max
Viscosity, 60°C, cP	T 316	30	60
Water, %	D95	–	0.2
Flash point, T.O.C., °F	T 79	95	–
Distillation test: [1]	T 78		
Distillate, percentage by volume of total distillate to 680°F			
to 437°F		–	35
to 500°F		30	75
to 600°F		75	95
Residue from distillation, volume %		50	–
Vacuum Recovery Test	Tex-555-C		
Residue from Vacuum Recovery Test, volume %		50	
Tests on Vacuum Recovered residue:			
Dynamic shear, G*/sinδ, 52°C, 10 rad/s, kPa	T 315	0.50	3.00
Solubility, %	T 44	99	–
Spot test	Tex-509-C	Neg.	

1 The distillation test is reserved for testing as needed or forensic purposes.

5.5. Table 6 Special-Use Cutback Asphalt

1. Delete Columns not used.

For SCM-I, the only remaining material in this table:

2. Replace Kinematic Viscosity with Rotational Viscosity.

- Replace AASHTO T 201, “Standard Method of Test for Kinematic Viscosity of Asphalts (Bitumens)” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”

- Additional data are being gathered to recommend a specification limit. Current suggestion is to leave the same limits. These are slightly different than cSt old units but are related by the specific gravity. Producers may make slight modifications to materials to meet these limits.

3. Keep Distillation as reserve test and not for routine testing.

- Add Vacuum Oven for standard testing of residue % and testing of residue.
- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

4. Replace Penetration with DSR at 46°C.

- Replace AASHTO T49, “Standard Method of Test for Penetration of Bituminous Materials” with AASHTO T315, “AASHTO T 315, “Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).”

5. Delete Polymer Content.

- While in the table, there are no requirements. This is a holdover from other deleted material.

6. Delete Ductility at 39.2°F.

- While in the table, there are no requirements. This is a holdover from other deleted material.

Table 6 of Item 300 after proposed changes:

Table 6

Special-Use Cutback Asphalt

Property	Test Procedure	Type–Grade	
		SCM I	
		Min	Max
Viscosity, 60°C, cP	T 316	500	1,000
Water, %	D95	–	0.2
Flash point, T.O.C., °F	T 79	175	–
Distillation test: [1]	T 78		
Distillate, percentage by volume of total distillate to 680°F			
to 437°F		–	–
to 500°F		–	0.5
to 600°F		20	60
Residue from distillation, volume %		76	–
Vacuum Recovery Test	Tex-554-C		
Residue from Vacuum Recovery Test, volume %		76	
Tests on Vacuum Recovered residue:			
Dynamic shear, G*/sinδ, 46°C, 10 rad/s, kPa [2]	T 315	0.5 [2]	2.0 [2]
Solubility, %	T 44	99	–
Spot test	Tex-509-C	Neg.	

1 The distillation test is reserved for testing as needed or forensic purposes.

2 Temperature and limits are place holders that need to be evaluated in the future.

5.6. Table 7 Emulsified Asphalt

1. Delete Columns not used.

2. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, “Standard Method of Test for Saybolt Viscosity” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”

- Additionally, perform all tests at 60°C, replacing testing at 25°C and 50°C.

3. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

4. Replace Residue Penetration with DSR at 58°C.

- Replace AASHTO T49, “Standard Method of Test for Penetration of Bituminous Materials” with AASHTO T315, “AASHTO T 315, “Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).”

5. Delete Residue Ductility.

- Delete AASHTO T51, “Standard Method of Test for Ductility of Asphalt Materials.”

6. Float Test – This test appears to capture an artifact of the distillation process. It is unclear whether this test adds additional value at this time and it should be investigated further.

Table 7 of Item 300 after proposed changes:

Table 7

Emulsified Asphalt

Property	Test Procedure	Type–Grade							
		Rapid-Setting		Medium-Setting		Slow-Setting			
		HFRS-2		MS-2		SS-1		SS-1H	
		Min	Max	Min	Max	Min	Max	Min	Max
Viscosity, 60°C, cP	T 316	320	850	210	650	40	210	40	210
Sieve test, %	T 59	–	0.1	–	0.1	–	0.1	–	0.1
Miscibility	T 59	–		–		Pass		Pass	
Cement mixing, %	T 59	–	–	–	–	–	2	–	2
Demulsibility, 35 mL of 0.02 N $CaCl_2$, %	T 59	50	–	–	30	–	–	–	–
Storage stability, 1 day, %	T 59	–	1	–	1	–	1	–	1
Freezing test, 3 cycles[1]	T 59	–		Pass		Pass		Pass	
Distillation test: [1]	T 59								
Residue by distillation, % by wt.		65	–	65	–	60	–	60	–
Oil distillate, % by volume of emulsion		–	0.5	–	0.5	–	0.5	–	0.5
Vacuum Recovery Test	Tex-555-C								
Residue by vacuum recovery, % by wt.		65		65		60		60	
Tests on vacuum recovered residue:									
Dynamic shear, G*/sinδ, 58°C, 10 rad/s, kPa [2]	T 315	0.5 [2]	2.0 [2]	0.5 [2]	2.0 [2]	0.5 [2]	2.0 [2]	0.5 [2]	2.0 [2]
Solubility, %	T 44	97.5	–	97.5	–	97.5	–	97.5	–
Float test, 140°F, sec.	T 50	1,200	–	–	–	–	–	–	–

1. The distillation test is reserved for testing as needed or forensic purposes.
2. Temperature and values are placeholders that need to be evaluated in the future.

5.7. Table 8 Cationic Emulsified Asphalt

1. Delete all empty columns.
2. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, “Standard Method of Test for Saybolt Viscosity” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”
- Additionally, perform all tests at 60°C, replacing testing at 25°C and 50°C.

3. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

4. Replace Residue Penetration with DSR at 58°C.

- Replace AASHTO T49, “Standard Method of Test for Penetration of Bituminous Materials” with AASHTO T315, “AASHTO T 315, “Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).”.

5. Delete Residue Ductility.

- Delete AASHTO T51, “Standard Method of Test for Ductility of Asphalt Materials.”

Table 8 of Item 300 after proposed changes:

Table 8

Cationic Emulsified Asphalt

Property	Test Procedure	Type–Grade							
		Rapid-Setting		Medium-Setting		Slow-Setting			
		CRS-2		CMS-2		CSS-1		CSS-1H	
		Min	Max	Min	Max	Min	Max	Min	Max
Viscosity, 60°C, cP	T 316	320	850	210	750	40	210	40	210
Sieve test, %	T 59	–	0.1	–	0.1	–	0.1	–	0.1
Cement mixing, %	T 59	–	–	–	–	–	2	–	2
Coating ability and water resistance:									
Dry aggregate/after spray	T 59	–		Good/Fair		–		–	
Wet aggregate/after spray		–		Fair/Fair		–		–	
Demulsibility, 35 mL of 0.8%, Sodium dioctyl sulfosuccinate, %	T 59	70	–	–	–	–	–	–	–
Storage stability, 1 day, %	T 59	–	1	–	1	–	1	–	1
Particle charge	T 59	Positive		Positive		Positive		Positive	
Distillation test: [1]									
Residue by distillation, % by wt.	T 59	65	–	65	–	60	–	60	–
Oil distillate, % by volume of emulsion		–	0.5	–	7	–	0.5	–	0.5
Vacuum Recovery Test	Tex-555-C								
Residue by vacuum recovery, % by wt.		65		65		60		60	
Tests on vacuum recovered residue:									
Dynamic shear, G*/sinδ, 58°C, 10 rad/s, kPa [2]	T 315	0.5 [2]	2.0 [2]	0.5 [2]	2.0 [2]	0.5 [2]	2.0 [2]	0.5 [2]	2.0 [2]
Solubility, %	T 44	97.5	–	97.5	–	97.5	–	97.5	–

1. The distillation test is reserved for testing as needed or forensic purposes.
2. Temperature and values are placeholders that need to be evaluated in the future.

5.8. Table 9 Polymer-Modified Emulsified Asphalt

1. Delete all empty columns.

Note: HFRS-2P is the only remaining material in the table.

2. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, "Standard Method of Test for Saybolt Viscosity" with AASHTO T 316, "Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer."

- Additionally, perform all tests at 60°C, replacing testing at 25°C and 50°C.

3. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, "Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven."

4. Replace Residue Penetration with DSR at 58°C.

- Replace AASHTO T49, "Standard Method of Test for Penetration of Bituminous Materials" with AASHTO T315, "AASHTO T 315, "Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)."

5. Replace Absolute Viscosity with DSR at 60°C or 64°C.

- Replace AASHTO T 202, "Standard Method of Test for Viscosity of Asphalts by Vacuum Capillary Viscometer" with AASHTO T 315, "Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)."

6. Replace Ductility and Elastic Recovery with MSCR Recovery.

- Replace AASHTO T51, "Standard Method of Test for Ductility of Asphalt Materials" and Tex-539-C "Measurement of Elastic Recovery of Tensile Deformation using a Ductilometer" with AASHTO T350 "Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)." This would measure the recovery at 0.1kPa, tested at the DSR test temperature.

- Additional data gathering is required to recommend specification limits.

7. Float Test – This test appears to capture an artifact of the distillation process. It is unclear whether this test adds additional value at this time and it should be investigated further.

Table 9 of Item 300 after proposed changes:

Table 9

Polymer-Modified Emulsified Asphalt

Property	Test Procedure	Type–Grade	
		Rapid-Setting	
		HFRS-2P	
		Min	Max
Viscosity, 60°C, cP	T 316	320	850
Sieve test, %	T 59	–	0.1
Demulsibility, 35 mL of 0.02 N $CaCl_2$, %	T 59	50	–
Storage stability, 1 day, %	T 59	–	1
Breaking index, g	Tex-542-C	–	–
Distillation test: [1, 2]	T 59		
Residue by distillation, % by wt.		65	–
Oil distillate, % by volume of emulsion		–	0.5
Vacuum Recovery Test	Tex-555-C		
Residue by vacuum recovery, % by wt.		65	
Tests on vacuum recovered residue:			
Polymer content, wt. % (solids basis)	Tex-533-C	3	–
Dynamic shear, G*/sinδ, 58°C, 10 rad/s, kPa [3]	T 315	0.5 [3]	2.0 [3]
MSCR Recovery, 0.1 kPa, DSR Temperature, % Min [3]	T350	30 [3]	
Solubility, %	T 44	97.5	–
Float test, 140°F, sec.	T 50	1200	–

1. The distillation test is reserved for testing as needed or forensic purposes.
2. Exception to T 59: Bring the temperature on the lower thermometer slowly to 350 ± 10°F. Maintain at this temperature for 20 min. Complete total distillation in 60 ± 5 min. from the first application of heat.
3. Temperature and values are placeholders that need to be evaluated in the future.

5.9. Table 10 Polymer-Modified Cationic Emulsified Asphalt

1. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, “Standard Method of Test for Saybolt Viscosity” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”

- Additionally, perform all tests at 60°C, replacing testing at 25°C and 50°C.

2. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

3. Delete Residue Penetration.

4. Replace Absolute Viscosity with DSR.

- Replace AASHTO T 202, “Standard Method of Test for Viscosity of Asphalts by Vacuum Capillary Viscometer” with AASHTO T 315, “Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).”

- Additional data gathering is required to recommend specification limits.

5. Replace Ductility and Elastic Recovery with MSCR Recovery.

- Replace AASHTO T51, “Standard Method of Test for Ductility of Asphalt Materials” and Tex-539-C “Measurement of Elastic Recovery of Tensile Deformation using a Ductilometer” with AASHTO T350 “Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).” This would measure the recovery at 0.1kPa, tested at the DSR test temperature.

- Additional data gathering is required to recommend specification limits.

6. Float Test – This test appears to capture an artifact of the distillation process. It is unclear whether this test adds additional value at this time and it should be investigated further.

Table 10 of Item 300 after proposed changes:

Table 10
Polymer-Modified Cationic Emulsified Asphalt

Property	Test Procedure	Rapid-Setting						Medium-Setting				Slow-Setting	
		CRS-2P		CHFRS-2P		CRS-2TR[4]		CMS-1P[3]		CMS-2P[3]		CSS-1P	
		Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Viscosity, 60°C, cP	T 316	320	850	210	850	320	1100	20	210	100	850	40	210
Sieve test, %	T 59	–	0.1	–	0.1	–	0.1	–	0.1	–	0.1	–	0.1
Demulsibility, 35 ml of 0.8% sodium dioctyl sulfosuccinate, %	T 59	70	–	60	–	40	–	–	–	–	–	–	–
Storage stability, 1 day, %	T 59	–	1	–	1	–	1	–	1	–	1	–	1
Breaking index, g	Tex-542-C	–	–	–	–	–	–	–	–	–	–	–	–
Particle charge	T 59	Positive		Positive		Positive		Positive		Positive		Positive	
Distillation test: [1, 2]													
Residue by distillation, % by wt.	T 59	65	–	65	–	65	–	30	–	60	–	62	–
Oil distillate, % by volume of emulsion		–	0.5	–	0.5	–	3	–	0.5	–	0.5	–	0.5
Vacuum Recovery Test Residue by vacuum recovery, % by weight	Tex-555-C	65	–	65	–	65	–	30	–	60	–	62	–
Tests on residue from vacuum recovery:													
Polymer content, wt. % (solids basis)	Tex-533-C	3	–	3	–	5	–	–	–	–	–	3	–
Dynamic shear, G*/sin(δ), Min, 1.00 kPa, Test temperature @ 10 rad/sec., °C [5]	T 315	64 [5]	–	64 [5]	–	64 [5]	–	64 [5]	–	64 [5]	–	64 [5]	–
MSCR Recovery, 0.1 kPa, DSR Temperature, % Min	T350	30	–	30	–	–	–	–	–	–	–	–	–
Nonrecoverable creep compliance of residue, 3.2 kPa, 52°C, kPa-1	T350	–	–	–	–	–	–	–	2.0	–	4.0	–	–
Float test, 140°F, sec.	T 50	–	–	1,800	–	–	–	–	–	–	–		
Solubility, %	T 44	97	–	95	–	98	–	–	–	–	–	97	–

1. The distillation test is reserved for testing as needed or forensic purposes.
2. Exception to T 59: Bring the temperature on the lower thermometer slowly to 350 ± 10°F. Maintain at this temperature for 20 min. Complete total distillation in 60 ± 5 min. from the first application of heat.
3. With all precertification samples of CMS-1P or CMS-2P, submit certified test reports showing the type and percent of rejuvenator and/or latex added. Submit samples of these raw materials if requested by the Engineer.
4. Modifier type is TR. Determined in accordance with Tex-553-C.
5. Temperature and values are placeholders that need to be evaluated in the future.

5.10. Table 10A Non-Tracking Tack Coat Emulsion

1. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, “Standard Method of Test for Saybolt Viscosity” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”

- Additionally, perform all tests at 60°C, replacing testing at 25°C.

2. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

3. Replace Residue Penetration with DSR at 82°C.

- Replace AASHTO T49, “Standard Method of Test for Penetration of Bituminous Materials” with AASHTO T315, “AASHTO T 315, “Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).”
- Delete Penetration.

Table 10A of Item 300 after proposed changes:

Table 10A

Non-Tracking Tack Coat Emulsion[1]

Property	Test Procedure	NT-HRE		NT-RRE		NT-SRE	
		Min	Max	Min	Max	Min	Max
Viscosity, 60°C, cP	T 316	30	–	30	–	20	210
Storage stability, 1 Day, %	T 59	–	1	–	1	–	1
Settlement, 5-day, %	T 59	–	5	–	5	–	5
Sieve test, %	T 59	–	0.3	–	0.3	–	0.1
Distillation test: [2, 3]	T 59						
Residue by distillation, % by wt.		50	–	58	–	50	–
Oil distillate, by volume of emulsion		–	1	–	1	–	1
Vacuum Recovery Test	Tex-554-C						
Residue by vacuum recovery, % by weight		50		58		50	
Test on residue from vacuum recovery:							
Dynamic shear, G*/sin(δ), 82°C, 10 rad/s, kPa [4]	T 315	1.0 [4]	–	1.0 [4]	–	1.0 [4]	–
Solubility, %	T 44	97.5		97.5		97.5	

1. These are emulsion-based TRAILs. Due to the hardness of the residue, these emulsions should be heated to 120–140° F prior to thorough mixing as the emulsion is being prepared for testing.
2. The distillation test is reserved for testing as needed or forensic purposes.
3. Exception to T 59: Bring the temperature on the lower thermometer slowly to 350 ± 10°F. Maintain at this temperature for 20 min. Complete total distillation in 60 ± 5 min. from the first application of heat.
4. Temperature and values are placeholders that need to be evaluated in the future.

5.11. Table 10B Spray Applied Underseal Membrane Polymer-Modified Emulsion (EBL)

This table only covers EBL.

1. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, “Standard Method of Test for Saybolt Viscosity” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”
- Additionally, perform test at 60°C, replacing testing at 25°C.
- Conditions and criteria/thresholds for different grades need to be developed.

2. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

3. Replace Residue Penetration with DSR.

- Replace AASHTO T49, “Standard Method of Test for Penetration of Bituminous Materials” with AASHTO T315, “AASHTO T 315, “Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).”

4. Replace Elastic Recovery with MSCR Recovery at temperature of DSR above.

Table 10B of Item 300 after proposed changes:

Table 10B

Spray Applied Underseal Membrane Polymer-Modified Emulsions (EBL)

Property	Test Procedure	Min	Max
Viscosity, 60°C, cP	T 316	40	210
Storage Stability [1], %	T 59	–	1
Demulsibility [2] Anionic emulsions — 35 ml of 0.02 N CaCl2, % Cationic emulsions — 35 ml 0.8% sodium dioctyl sulfosuccinate, %	T 59	55	–
Sieve Test [3], %	T 59	–	0.05
Distillation Test: [4, 5] Residue by distillation, % by wt. Oil portion of distillate, % by vol.	T 59	63 –	– 0.5
Vacuum Recovery Test Residue by vacuum recovery, % by weight	Tex-555-C	63	–
Test on residue from vacuum recovery: MSCR Recovery, 0.1 kPa, 58C, % Min Dynamic shear, G*/sinδ, 64°C, 10 rad/s, kPa	T350 T 49	30 0.5	– 2.0

1. After standing undisturbed for 24 hr., the surface must be smooth, must not exhibit a white or milky colored substance, and must be a homogeneous color throughout.
2. Material must meet demulsibility test for emulsions.
3. May be required by the Engineer only when the emulsion cannot be easily applied in the field.
4. The distillation test is reserved for testing as needed or forensic purposes.
5. The temperature on the lower thermometer should be brought slowly to 350 ± 10°F and maintained at this temperature for 20 min. The total distillation should be completed in 60 ± 5 min. from the first application of heat.
6. Temperature and values are placeholders that need to be evaluated in the future.

5.12. Table 10C Full-Depth Reclamation Emulsion (FDR EM)

This table only covers FDR EM materials.

1. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, “Standard Method of Test for Saybolt Viscosity” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”
- Additionally, perform all tests at 60°C, replacing testing at 25°C.

2. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

3. Replace Residue Penetration with DSR at 64°C.

- Replace AASHTO T49, "Standard Method of Test for Penetration of Bituminous Materials" with AASHTO T315, "AASHTO T 315, "Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)."

Table 10C of Item 300 after proposed changes:

Table 10C

Full-Depth Reclamation Emulsion (FDR EM)

Property	Test Procedure	Standard Yield (SY)		High Yield (HY)[3]	
		Min	Max	Min	Max
Viscosity, 60°C, cP	T 316	40	210	40	210
Sieve test, %	T 59	–	0.1	–	0.1
Cement mixing, %	T 59	–	2	–	2
% Storage stability, 1 day, %	T 59	–	1	–	1
Distillation test: [1, 2]	T 59				
Residue by distillation, % by wt.		60	–	63	–
Oil portion of distillate, % by vol.		–	0.5	–	0.5
Vacuum Recovery Test	Tex-555-C				
Residue by vacuum recovery, % by weight		60	–	63	–
Test on residue from vacuum recovery:					
Dynamic shear, G*/sinδ, 64°C, 10 rad/s, kPa [4]	T 315	0.5 [4]	2.0 [4]	0.5 [4]	2.0 [4]

1. The distillation test is reserved for testing as needed or forensic purposes.

2. The temperature on the lower thermometer should be brought slowly to 350 ± 10°F and maintained at this temperature for 20 min. The total distillation should be completed in 60 ± 5 min. from the first application of heat.

3. Provide a manufacturer's certificate of analysis (COA) with the type and percent of rejuvenator added.

4. Temperature and values are placeholders that need to be evaluated in the future.

5.13. Table 11 Specialty Emulsions

1. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, "Standard Method of Test for Saybolt Viscosity" with AASHTO T 316, "Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer."

- Additionally, perform all tests at 60°C, replacing testing at 25°C and 50°C.

2. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

3. For AEP, Vacuum Oven replaces both the emulsion and cutback distillation procedures.

4. For PCE, Replace Evaporation with Vacuum Oven.

- Replace T59, “Standard Method of Test for Emulsified Asphalts, Section 7 - Emulsified Asphalt Residue by Evaporation” with Tex-555-C, “ Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

5. Replace Kinematic and Absolute Viscosity at 140°F with Rotational Viscosity at 140°F.

- Replace AASHTO T 201, “Standard Method of Test for Kinematic Viscosity of Asphalts (Bitumens)” and AASHTO T 202, “Standard Method of Test for Viscosity of Asphalts by Vacuum Capillary Viscometer” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”

- Additional data gathering is required to recommend specification limits.

6. Replace Float with Rotational Viscosity at 140°F.

- For AE-P, replace Float Test with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.” Threshold needs to be developed. Suggest 300-1200 Pa-s like MC-30.

Table 11 of Item 300 after proposed changes:

Table 11

Specialty Emulsions

Property	Test Procedure	Type–Grade					
		Medium-Setting				Slow-Setting	
		AE–P		EAP&T		PCE[1]	
		Min	Max	Min	Max	Min	Max
Viscosity, 60°C, cP	T 316	30	320	20	210	20	210
Sieve test, %	T 59	–	0.1	–	0.1	–	0.1
Miscibility[2]	T 59	–		Pass		Pass	
Demulsibility, 35 mL of 0.10 N $CaCl_2$, %	T 59	–	70	–	–	–	–
Storage stability, 1 day, %	T 59	–	1	–	1	–	–
Particle size,[5] % by volume < 2.5 µm	Tex-238-F [3]	–	–	90	–	90	–
Asphalt emulsion distillation to 500°F followed by Cutback asphalt distillation of residue to 680°F: [6]	T 59 & T 78						
Residue after both distillations, % by wt.		40	–	–	–	–	–
Total oil distillate from both distillations, % by volume of emulsion		25	40	–	–	–	–
Residue by distillation, % by wt. [6]	T 59	–	–	60	–	–	–
Residue by evaporation, % by wt. [4, 6]	T 59	–	–	–	–	60	–
Vacuum Recovery Test	Tex-554-C						
Residue by vacuum recovery, % by wt.		40	–	60	–	60	–
Test on residue from vacuum recovery:							
Viscosity, 60°C, Pa·s	T 316	300	1200	80	–	10	35
Flash point C.O.C., °F	T 48	–	–	–	–	400	–
Solubility, %	T 44	97.5	–	–	–	–	–

1. Supply with each shipment of PCE:
   - a copy of a lab report from an approved analytical lab, signed by a lab official, indicating the PCE formulation does not meet any characteristics of a Resource Conservation Recovery Act (RCRA) hazardous waste;
   - a certification from the producer that the formulation supplied does not differ from the one tested and that no listed RCRA hazardous wastes or Polychlorinated Biphenyls (PCBs) have been mixed with the product; and
   - a Safety Data Sheet.
2. Exception to T 59: In dilution, use 350 mL of distilled or deionized water and a 1,000-mL beaker.
3. Use Tex-238-F, beginning at "Particle Size Analysis by Laser Diffraction," with distilled or deionized water as a medium and no dispersant, or use another approved method.
4. Exception to T 59: Leave sample in the oven until foaming ceases, then cool and weigh.
5. PCE must meet either the kinematic viscosity requirement or the particle size requirement.
6. The distillations and evaporation testing are reserved for testing as needed or forensic purposes. Vacuum recovery is standard test.

5.14. Table 11A Hard Residue Surface Sealant (HRSS)

1. This table only covers HRSS material. This material may be deleted depending on the current use history.

2. Replace Krebs Viscosity with Rotational Viscosity.

- Replace ASTM D562, “Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”

3. Replace Evaporation with Vacuum Oven.

- Replace ASTM D 2939, “Standard Test Methods for Emulsified Bitumens Used as Protective Coatings – Section 8, Residue by Evaporation” with Tex-555-C, “ Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

4. Replace Softening Point (Tex-505-C) and Penetration with DSR.

- Replace both with DSR at an elevated temperature, possibly 82°C. This has to be investigated.

- Additional data gathering is required to recommend specification limits.

Table 11A of Item 300 after proposed changes:

Table 11A

Hard Residue Surface Sealant (HRSS)

Property	Test Procedure	Min	Max
Viscosity, 60°C, cP	T 316	140	750
Uniformity	D 2939	Pass[2]	
Resistance to heat	D 2939	Pass[3]	
Resistance to water	D 2939	Pass[4]	
Wet flow, mm	D 2939	--	0
Resistance to Kerosene (optional)[5]	D 2939	Pass[6]	
Ultraviolet exposure, UVA-340, 0.77 W/m^2, 50°C chamber, 8 hr. UV lamp, 5 min. spray, 3 hr. 55 min. condensation, 1,000 hr. total exposure[7]	G 154	Pass[8]	
Abrasion loss, 1.6 mm thickness, liquid only, %	ISSA TB-100	--	1
Vacuum Recovery Test	Tex-555-C		
Residue by vacuum recovery, % by weight		33	--
Tests on residue from vacuum recovery:			
Dynamic shear, G*/sinδ, 82°C, 10 rad/s, MPa [9]	T 315	0.5 [9]	2.0 [9]
Flash point, Cleveland open cup, °F	T 48	500	--
Tests on base asphalt before emulsification			
Solubility, %	T 44	98	--

1. Cure the emulsion in the softening point ring in a 200 ± 5°F oven for 2 hr.
2. Product must be homogenous and show no separation or coagulation that cannot be overcome by moderate stirring.
3. No sagging or slippage of film beyond the initial reference line.
4. No blistering or re-emulsification.
5. Recommended for airport applications or where fuel resistance is desired.
6. No absorption of Kerosene into the clay tile past the sealer film. Note sealer surface condition and loss of adhesion.
7. Other exposure cycles with similar levels of irradiation and conditions may be used with Department approval.
8. No cracking, chipping, surface distortion, or loss of adhesion. No color fading or lightening.
9. Temperature and values are placeholders that need to be evaluated in the future.

5.15. Table 12 Diluted CSS-1H

This table only covers diluted CSS-1H.

1. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, “Standard Method of Test for Saybolt Viscosity” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”

- Additionally, perform all tests at 60°C, replacing testing at 25°C.
- For this material, this requirement is only to report. There is no specification limit as only a maximum makes sense.

2. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

3. Replace Residue Penetration with DSR at 64°C.

- Replace AASHTO T49, “Standard Method of Test for Penetration of Bituminous Materials” with AASHTO T315, “AASHTO T 315, “Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR).”
- Additional data gathering is required to recommend specification limits.

4. Delete Residue Ductility.

- Delete AASHTO T51, “Standard Method of Test for Ductility of Asphalt Materials.”

Table 12 of Item 300 after proposed changes:

Table 12

Diluted CSS-1H

Property	Test Procedure	Type-Grade					
		Diluted Slow-Setting					
		CSS-1H 50/50		CSS-1H 40/60		CSS-1H 30/70	
		Min	Max	Min	Max	Min	Max
Viscosity, 60°C, cP	T 316	Report Only		Report Only		Report Only	
Distillation test: [1]							
Residue by distillation, % by wt.	T 59	30	–	24	–	18	–
Oil distillate, % by volume of emulsion		–	0.5	–	0.5	–	0.5
Vacuum Recovery Test	Tex-554-C						
Residue by vacuum recovery, % by weight		30		24		18	
Tests on residue from vacuum recovery:							
Dynamic shear, G*/sinδ, 64°C, 10 rad/s, kPa [2]	T 315	0.5 [2]	2.0 [2]	0.5 [2]	2.0 [2]	0.5 [2]	2.0 [2]
Solubility, %	T 44	97.5	–	97.5	–	97.5	–

1. The distillation test is reserved for testing as needed or forensic purposes.
2. Temperature and values are placeholders that need to be evaluated in the future.

5.16. Table 12A Diluted AE-P

This table only covers diluted AE-P.

1. Replace Saybolt Viscosity with Rotational Viscosity and change test temperature.

- Replace T72, “Standard Method of Test for Saybolt Viscosity” with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”

- Additionally, perform all tests at 60°C, replacing testing at 25°C.

- For this material, this requirement is only to report. There is no specification limit as only a maximum makes sense.

2. Replace Distillation with Vacuum Oven for residue % and testing residue.

- Add Tex-555-C, “Recovery of Asphalt Residue from Asphalt Emulsion and Cutback Asphalt by Vacuum Oven.”

3. Replace Float with Rotational Viscosity at 140°F.

- For AE-P, replace Float Test with AASHTO T 316, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer.”

- Additional data gathering is required to recommend specification limits.
- Suggest 300-1200 Pa-s like MC-30.

Table 12A of Item 300 after proposed changes:

Table 12A

Diluted AE-P

Property	Test Procedure	Type–Grade					
		Diluted Slow-Setting					
		AE-P 50/50		AE-P 40/60		AE-P 30/70	
		Min	Max	Min	Min	Max	Min
Viscosity, 60°C, cP	T 316	Report Only		Report Only		Report Only	
Asphalt emulsion distillation to 500°F followed by Cutback asphalt distillation of residue to 680°F: [1]	T 59 & T 78						
Residue after both distillations, % by wt.		20	–	16	–	12	–
Total oil distillate from both distillations, % by volume of emulsion		12.5	20	10	16	7.5	12
Vacuum Recovery Test	Tex-554-C						
Residue by vacuum recovery, % by wt.		20	–	16	–	12	–
Tests on residue after vacuum recovery:							
Solubility, %	T 44	97.5	–	97.5	–	97.5	–
Viscosity, 60°C, Pa·s	T 316	300	1200	300	1200	3000	1200

1. The distillations and evaporation testing are reserved for testing as needed or forensic purposes. Vacuum recovery is the standard test. If distillation is required, tests on vacuum recovery apply.

5.17. Table 13 Recycling Agent and Emulsified Recycling Agent

1. Since this specification has no requirements on recycling and rejuvenation, we suggest deleting the entire table.

5.18. Table 14 CRM Gradations

No changes.

5.19. Table 15 Polymer-Modified Asphalt-Emulsion Crack Sealer

1. Change Rotational Viscosity Test Procedure.

- Change Rotational Viscosity test procedure from D2196, Method A to AASHTO T 316, "Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer."

- This material is used at room temperature, so it makes sense to keep the test temperature at 77°F as in the current requirement.

2. Replace Penetration at 77°F, Softening Point, and Ductility at 39.2°F with DSR and MSCR Recovery.

Table 15 of Item 300 after proposed changes:

Table 15

Polymer-Modified Asphalt-Emulsion Crack Sealer

Property	Test Procedure	Min	Max
Rotational viscosity, 77°F, cP	D 2196, Method A	10,000	25,000
Sieve test, %	T 59	–	0.1
Storage stability, 1 day, %	T 59	–	1
Vacuum Recovery Test Residue by vacuum recovery, % by weight	Tex-554-C	65	–
Tests on residue from vacuum recovery:			
Dynamic shear, G*/sin(d), Min, 1.00 kPa, Test temperature @ 10 rad/sec., °C [1]	T 315	70 [1]	
MSCR Recovery, 0.1 kPa, DSR Temperature, % Min	T 350	20	

1. Temperature and values are placeholders that need to be evaluated in the future.

5.20. Table 16 Asphalt-Rubber Crack Sealer

This table is for Rubber-Asphalt Crack Sealer.

1. Penetration and Softening Point can be replaced by DSR; however, there needs to be investigation into how the DSR functions with this material that has granulated rubber particles in it. These Pen tests are cone penetrometer which is not a standard penetration. Suggest leaving this the same, unless additional data is gathered to use DSR.

- Additional data gathering is required to recommend specification limits.

Table 16 of Item 300 after proposed changes:

Table 16

Rubber-Asphalt Crack Sealer

Property		Class A	Class B

	Test Procedure	Min	Max	Min	Max
CRM content, Grade A or B, % by wt.	Tex-544-C	22	26	–	–
CRM content, Grade B, % by wt.	Tex-544-C	–	–	13	17
Virgin rubber content,[1] % by wt.		–	–	2	–
Flash point,[2] C.O.C., °F	T 48	400	–	400	–
Penetration,[3] 77°F, 150 g, 5 sec.	T 49	30	50	30	50
Penetration,[3] 32°F, 200 g, 60 sec.	T 49	12	–	12	–
Softening point, °F	T 53	–	–	170	–
Bond Test, non-immersed, 0.5 in specimen, 50% extension, 3 cycles, 20°F[4]	D5329	–		Pass	

1. Provide certification that the Min % virgin rubber was added.
2. Agitate the sealing compound with a 3/8- to 1/2-in. (9.5- to 12.7-mm) wide, square-end metal spatula to bring the material on the bottom of the cup to the surface (i.e., turn the material over) before passing the test flame over the cup. Start at one side of the thermometer, move around to the other, and then return to the starting point using 8 to 10 rapid circular strokes. Accomplish agitation in 3 to 4 sec. Pass the test flame over the cup immediately after stirring is completed.
3. Exception to T 49: Substitute the cone specified in D 217 for the penetration needle.
4. Allow no crack in the crack sealing materials or break in the bond between the sealer and the mortar blocks over 1/4 in. deep for any specimen after completion of the test.

5.21. Table 17 A-R Binders

This table is for Asphalt-Rubber Binders.

1. This specification is based on the ASTM specifications for A-R Binders and should be considered before modification.

2. If modified, Penetration and Softening Point could be replaced by DSR, and Resilience could be replaced by MSCR recovery. Tests on RTFOT residue for retained penetration ratio may be able to be measured by a DSR aging ratio or max DSR.

2. This would require more work. Conditions and criteria/thresholds for different grades need to be developed.

Table 17 of Item 300 after proposed changes:

Table 17

A-R Binders

Property	Test Procedure	Binder Type					
		Type I		Type II		Type III	
		Min	Max	Min	Max	Min	Max
Apparent viscosity, 347°F, cP	D2196, Method A	1,500	5,000	1,500	5,000	1,500	5,000
Penetration, 77°F, 100 g, 5 sec.	T 49	25	75	25	75	50	100
Penetration, 39.2°F, 200 g, 60 sec.	T 49	10	–	15	–	25	–
Softening point, °F	T 53	135	–	130	–	125	–
Resilience, 77°F, %	D5329	25	–	20	–	10	–
Flash point, C.O.C., °F	T 48	450	–	450	–	450	–
Tests on residue from RTFOT: Retained penetration ratio, 39.2°F, 200 g, 60 sec., % of original	T 240 T 49	75	–	75	–	75	–

5.22. Table 18 Performance-Graded Binders

This table is for PG binders.

1. Retain MSCR Recovery.
2. Add 8mm, Low Temperature DSR as surrogate for BBR.

- Add Tex-554-C, “Low Temperature DSR as a Surrogate for S and M-value” (actual title to be determined). This would be 8mm DSR at PG Low + 10°C with phase angle > 22.0 degrees and G* < 220,000 kPa. Add a footnote that a binder must meet either low temp DSR requirements or BBR S and m-value.

3. Replace ER with Poker Chip or add Poker Chip.

- Replace Tex-539-C “Measurement of Elastic Recovery of Tensile Deformation using a Ductilometer” with AASHTO TP 150, “Poker Chip Test of Asphalt Binder”. Use the limits of:
    - 70-22, 76-22, and 82-16 = 400%
    - 64-28, 70-28, and 82-22 = 600%
    - 64-34, 70-34, 76-28, 82-28 = 800%
    - 76-34 = 1000%

4. Delete Direct Tension.

- Delete AASHTO T 314, "Standard Method of Test for Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT)." It is not currently performed, and the equipment has not proven to be robust.

Table 18 of Item 300 after proposed changes (and according changes enacted in SP 300-003):

Table 18
Performance-Graded Binders

Property and Test Method	Performance Grade																		
	PG 58			PG 64				PG 70				PG 76				PG 82			
	-22	-28	-34	-16	-22	-28	-34	-16	-22	-28	-34	-16	-22	-28	-34	-16	-22	-28	
Average 7-day max pavement design temperature, °C[1]	58			64				70				76				82			
Min pavement design temperature, °C[1]	-22	-28	-34	-16	-22	-28	-34	-16	-22	-28	-34	-16	-22	-28	-34	-16	-22	-28	
Original Binder																			
Flash point, T 48, Min, °C	230																		
Viscosity, T 316[4, 3]: Max, 3.0 Pa.s, test temperature, °C	135																		
Dynamic shear, T 315[4]: G*/sin(δ), Min, 1.00 kPa, Max, 2.00 kPa[5], Test temperature @ 10 rad/sec., °C	58			64				70				76				82			
Elastic recovery, D6084, 50°F, % Min[6]	–	–	30	–	–	30	50	–	30	50	60	30	50	60	70	50	60	70	
Poker Chip, TP 150, Ductility, Min	–	–	–	–	–	600	800	–	400	600	800	–	400	800	1000	400	600	800	
Rolling Thin-Film Oven (RTFO) (T 240)																			
Mass change, T 240, Max, %	1.0																		
Dynamic shear, T 315: G*/sin(δ), Min, 2.20 kPa, Max, 5.00 kPa[5], Test temperature @ 10 rad/sec., °C	58			64				70				76				82			
MSCR, T350, Recovery, 0.1 kPa, High Temperature, % Min[6]	–	–	20	–	–	20	30	–	20	30	40	20	30	40	50	30	40	50	
Pressure Aging Vessel (PAV) Residue (R 28)																			
PAV aging temperature, °C	100																		
Dynamic shear, T 315 G*.sin(δ), Max, 5,000 kPa (Max, 6,000 kPa for δ ≥42°) Test temperature @ 10 rad/sec., °C	25	22	19	28	25	22	19	28	25	22	19	28	25	22	19	28	25	22	
Dynamic shear, Tex-554-C[7]: G*, Max, 220 MPa δ, min. 22° (Test temperature @ 0.2 rad/sec., °C)	-12	-18	-24	-6	-12	-18	-24	-6	-12	-18	-24	-6	-12	-18	-24	-6	-12	-18	
Creep stiffness, T 313[8]: S, max, 300 MPa, m-value, Min, 0.300 Test temperature @ 60 sec., °C	-12	-18	-24	-6	-12	-18	-24	-6	-12	-18	-24	-6	-12	-18	-24	-6	-12	-18	

1. Pavement temperatures are estimated from air temperatures and using an algorithm contained in a Department-supplied computer program, may be provided by the Department, or may be obtained following the procedures outlined in AASHTO MP 323 and R 25.
2. This requirement may be waived at the Department's discretion if the supplier warrants that the asphalt binder can be adequately pumped, mixed, and compacted at temperatures that meet all applicable safety, environmental, and constructability requirements. At test temperatures where the binder is a Newtonian fluid, any suitable standard means of viscosity measurement may be used, including capillary (T 201 or T 202) or rotational viscometry (T 316).
3. Viscosity at 135°C is an indicator of mixing and compaction temperatures that can be expected in the lab and field. High values may indicate high mixing and compaction temperatures. Additionally, significant variation can occur from batch to batch. Contractors should be aware that variation could significantly impact their mixing and compaction operations. Contractors are therefore responsible for addressing any constructability issues that may arise.
4. For quality control of unmodified asphalt binder production, measurement of the viscosity of the original asphalt binder may be substituted for dynamic shear measurements of G*/sin(δ) at test temperatures where the asphalt is a Newtonian fluid. Any suitable standard means of viscosity measurement may be used, including capillary (T 201 or T 202) or rotational viscometry (T 316).
5. Max values for unaged and RTFO-aged dynamic shear apply only to materials used as substitute binders, as described in Item 341, "Dense-Graded Hot-Mix Asphalt," and Item 344, "Superpave Mixtures."
6. Elastic recovery (D6084) is not required unless MSCR (T 350) is less than the Min % recovery. Elastic recovery will be used for the acceptance criteria in this instance.
7. Bending beam rheometer, BBR (T313) is not required unless the low. temp. DSR (Tex-554-C) do not meet the afore specified requirements. BBR will be used for the acceptance criteria in this instance.
8. Silicone beam molds, as described in AASHTO TP 1-93, are acceptable for use.
9. Temperature and values are placeholders that need to be evaluated in the future.

Chapter 6. Evaluation of New Generation of Aging Protocol

6.1. Evaluating the Accelerated Long-term Aging Protocol for Asphalt Binder

6.1.1. Goal

Currently, asphalt binder aged in a pressure aging vessel with 3.2 mm film thickness (50 ± 0.5 g) at 100°C for 20 hrs. (PAV20) is used to simulate long-term aging in the field. However, it was reported that the PAV20, at best, simulates the aging that occurs during the first four to eight years of pavement service life [98]. A recently completed national study-NCHRP 9-61 recommended a 40-hr. PAV (or PAV40) aging procedure to simulate approximately ten years of field aging. Additionally, NCHRP 9-61 also recommended an alternative long-term aging procedure: PAV20 with a much thinner film: 0.8 mm, which can also simulate the near-surface field aging of ten years within a 20-hr. period. The main goal of this exercise is to:

- Evaluate the feasibility of using PAV20 with a 0.8 mm film thickness (Thin Film PAV20) in lieu of PAV40 as a more severe long-term aging procedures for asphalt binder and to propose a more practical test protocol for Thin Film PAV20.
- Evaluate the impact of the Thin Film PAV20 and PAV40 aging procedures on asphalt binder properties.

6.1.2. Scope

The proposed method was evaluated using asphalt binders with PG grades of PG64-22, PG70-22, PG76-22, PG70-28, and PG76-22.

6.1.3. Evaluation

6.1.3.1. Aging protocol

Four laboratory aging protocols were employed, including Rolling Thin-Film Oven (RTFO) to simulate short-term aging of asphalt binders, and PAV20, PAV40, and Thin Film PAV20 to simulate long-term aging.

6.1.3.1.1. RTFO, PAV20, and PAV40

The RTFO aging method is intended to simulate short-term aging of the binder during the mixture production at the asphalt mix plant, the storage and transport to the construction site, and compaction at the site. The test involves a moving film of asphalt binder in an open bottle placed in a rotating circular rack in an oven for 85 minutes at 163℃ with heated air blowing into each bottle at the rate of 4000 mL/min at the lowest point of travel. In this study, the AASHTO T 240 (2023) procedure was followed for the RTFO aging process.

The PAV20 and PAV40 aging methods are intended to simulate long-term aging of the binder experienced through the service life of the pavement. The oxidation that the binder will experience is simulated by placing 50 g of RTFO-aged binder as a Thin Film in a steel pan. The pan is placed in the PAV under 2.1 MPa of pressure for 20 hours or 40 hours at the temperature specified in AASHTO M 320 (normally 100℃). In this study, the AASHTO R 28 (2021) procedure was followed for the long-term aging process.

6.1.3.1.2. Modified Thin Film PAV20

The Thin Film PAV20 aging protocol was proposed in NCHRP 9-61 (2021) to simulate the ten years of field aging at a depth of 0.75 in (19 mm) for the pavements. To achieve a film thickness of 0.8 mm, the mass of binder in each PAV pan is reduced to 12.5 ± 0.5 g. In addition, to ensure a uniform film distribution, the pans are conditioned in an airtight oven at 135 °C under a nitrogen atmosphere prior to PAV aging, allowing the binder to flow and cover the flat portion of the pan. The pans are then placed in a leveled PAV and conditioned under standard PAV aging conditions for 20 hours. The PAV-conditioned residue is then vacuum degassed before binder testing.

However, in preliminary testing conducted in this study, the procedure revealed that the main challenge of Thin Film PAV20 lies in maintaining continuous and uniform film thickness. Unmodified binders such as PG64-22 were able to form a uniform film consistently, but modified binders, particularly PG76-22, often failed to fully cover the pan surface after the sample was placed in the oven. This issue may be related to the flatness of the PAV pan and the vacuum oven, as well as to the heating temperature and duration used in the vacuum oven. It was also observed that binder flow typically ceases after around 30 minutes of conditioning in the vacuum oven. Additionally, it was found that using commercially available stainless-steel pans with a thickness of 1.0 mm instead of the 0.635 mm required in R 28 helps prevent warping during high-temperature conditioning and facilitates the formation of a uniform asphalt film. Based on these experiences, and after a series of modifications and trials, a modified Thin Film PAV20 aging protocol is proposed as described below:

1. Before each run, verify the flatness of the pans in the pan holder when installed in the vacuum oven and in the PAV using a precision machinist level. Check flatness along

perpendicular axes as shown in Figure 6.1. Adjust as necessary to achieve a level plane within 0.025 degrees.

Figure 6.1 Leveling calibration.

2. Before each run, preheat the vacuum oven with the pan holder without the pans at 163°C and maintain the temperature for a minimum of 12 hours to ensure temperature consistency and uniformity.

3. Condition the asphalt binder in accordance with T 240 (RTFO).

4. After combining the RTFO residue into a single container and blending as specified in T 240, perform one of the three following actions.

    a) Pour the hot residue directly into the stainless-steel pans for immediate conditioning in the PAV beginning at Step 5 below; or

    b) Pour the hot residue into the stainless-steel pans according to Steps 5 and 6, then cover and set aside for conditioning at a later time beginning at Step 7; or

    c) Allow the residue to cool in the single container for conditioning at a later time beginning at Step 5. If the residue is stored in a single container, heat the residue, stir gently, and pour the heated residue into the pans.

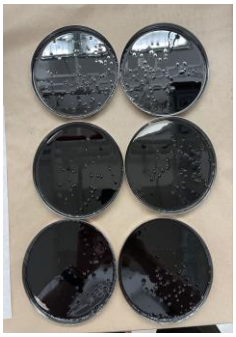
5. Preheat the PAV pans (preferably 1 mm thick rather than the 0.635 mm specified in R 28) in a 163 °C oven for ten minutes to facilitate asphalt flow and distribution.

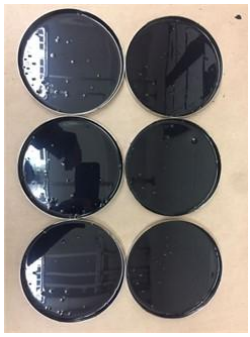
6. Place each stainless-steel pan on the balance and add 12.5 ± 0.1 g of RTFO residue to each pan. This amount of binder will yield a film thickness of approximately 0.8 mm.

7. After RTFO residue has been added to all pans, remove the pan holder from the vacuum oven and load it with the pans having 12.5 g of RTFO residue. Place the pan holder with loaded pans into the 163°C vacuum oven and close the vacuum oven.

8. Using the vacuum system to reduce the pressure in the vacuum chamber to 15 ± 2.5 kPa absolute in 2.5 minutes or less and then introduce nitrogen through the vent port. Condition the pans at 163°C for 30 ± 5 minutes.
9. Open the vacuum oven and remove the pans from vacuum chamber. It is acceptable to manually and uniformly adjust the angle of pans immediately after removal in order to promote a more uniform distribution of the binder.
10. Place the pans into the PAV and close the PAV.
11. Start the standard PAV20 aging according to R 28.
12. Open the PAV, remove the PAV pans and transfer them to an oven set at 170°C for ten minutes, and then immediately scrape it into containers.
13. Transfer the containers to a vacuum degassing oven and start the vacuum degassing procedure.
14. Remove the containers from the vacuum degassing oven and gently stir the binder to blend the residue.
15. Prepare test specimens directly from the residue in the containers; subdivide the residues into smaller containers for future testing or set the containers aside for future testing.

The typical binder distribution conditions after PAV20 are shown in Figure 6.2:


PG64-22


PG70-22


PG76-22


PG64-28

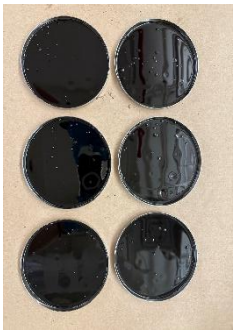

PG70-28

Figure 6.2 The typical binder distribution conditions after PAV20.

6.1.3.2. Method and tests

6.1.3.2.1. DSR test

Dynamic shear rheometer (DSR) was used for performance testing to compare the rheological properties of binders subjected to different aging protocols. The temperature sweep test was used to test binders at high and intermediate PG temperatures. The 8 mm DSR test (proposed replacement for BBR) was used to test binders at low PG temperature (not applicable for RTFO binder). The parameter G* and δ from the last five cycles was averaged. Both parameters were used as the rheological indicator. RTFO, PAV20, and PAV40 were tested with two replicates, while thin-film PAV20 was tested with three replicates. Error bars represent half the range between two replicates and the standard deviation for three replicates. This approach ensures a consistent representation of variability across different sample sizes, given the limited number of replicates. The DSR test procedures were summarized below, and the corresponding testing temperatures for different binders were shown in Table 6.1.

DSR Procedure

1. Condition the sample at the high PG temperature: Oscillate at 10% strain rate, 10 rad/s for ten cycles.
2. Condition the sample at the intermediate PG temperature: Oscillate at 1% strain rate, 10 rad/s for ten cycles.
3. Condition the sample at the low PG temperature: Oscillate at 0.1% strain rate, 0.2 rad/s for ten cycles.

Table 6.1 DSR testing temperatures for different binders.

Binder	High PG T (°C)	Intermediate PG T (°C)	Low PG T (°C)
PG 64-22	64	25	-12
PG 70-22	70	25	-12
PG 76-22	76	25	-12
PG 64-28	64	22	-18
PG 70-28	70	22	-18

6.1.3.2.2. Bending Beam Rheometer (BBR) test

BBR was used to measure the creep stiffness (*S*) and *m*-value (slope of the stiffness versus temperature log-log plot) at 60 seconds, which can be used to evaluate the binder's ability to resist stress and relax internal stresses to prevent thermal cracking. The device used for this study was the Cannon TE-BBR (Thermoelectric Bending Beam Rheometer), and the test was performed according to AASHTO T313 (2022), using a binder beam with a length of 127.0 mm, a height of 12.5 mm, and a thickness of 6.255 mm. The load applied has a magnitude of 980 mN, and the deflection response of the beam was measured upon application of the load. All BBR tests were conducted at the low PG temperature as shown in Table 6.1. Two replicates were conducted, and error bars were represented by half the range between the two replicates.

6.1.3.2.3. Poker-chip test

The Poker-chip test was used to compare the cracking performance between binders subjected to different aging protocols. The strength and ductility parameters obtained from the poker-chip test were adopted. The sample preparation procedures and detailed experimental protocols are described in the preceding sections. Two replicates were evaluated, and error bars were represented by half the range between two replicates.

6.1.3.2.4. Fourier-transform infrared (FTIR) spectroscopy

FTIR spectroscopy was utilized to evaluate the chemical aging condition of asphalt binders with different aging protocols. The attenuated total reflection geometry (ATR) mode was used. The FTIR spectra of each asphalt binder was recorded over a wavenumber range of 600 cm^{-1} to 4000 cm^{-1}. The absorbance areas of two oxidation-related bands, the carbonyl (C=O) and sulfoxide (S=O) functional groups, were calculated. The areas under the C=O absorption band (1666–1746 cm^{-1}), the S=O absorption band (944–1066 cm^{-1}), and the denominator region (1350–1525 cm^{-1}) were determined using full baseline integration. The carbonyl index (C=O index) and sulfoxide index (S=O index) were calculated as the ratio of the corresponding absorption area to the denominator area. Specifically, the C=O index was defined as the ratio of the C=O band area (1666–1746 cm^{-1}) to the area of the 1350–1525 cm^{-1} region, and the S=O index was defined as the ratio of the S=O band area (944–1066 cm^{-1}) to the same denominator area. Prior to each measurement, a background spectrum was collected using a clean and empty ATR crystal to ensure spectral accuracy. For each binder, four replicates were conducted, and the average values were reported. Standard deviations were calculated and presented as error bars to reflect data variability.

6.1.3.3. Materials

A total of eight binders with a variety of PG binders from different suppliers were used for the exercise. The specific supplier names were abbreviated, and the binder list is as follows:

• PG64-22 A

• PG64-22 H

• PG70-22 C

• PG70-22 V

• PG76-22 M

• PG76-22 W

• PG64-28 J

• PG70-28 E

6.1.4. Results and Discussion

6.1.4.1. DSR test results

Figure 6.3 to Figure 6.5 present the DSR parameters of eight binders tested at high-, intermediate-, and low-PG temperatures after different aging protocols. Overall, under the conventional aging sequence from unaged to PAV40, the G* gradually increases while δ decreases, which aligns with the commonly observed rheological behavior of asphalt binders. This behavior is attributed to oxidative aging, which enhances binder stiffness and reduces its viscous response.

As to the Thin Film PAV20, it is observed that under three temperatures, all binders exhibit significantly higher G* and lower δ compared to those aged under standard PAV20. Moreover, Thin Film PAV20 consistently produces rheological parameters comparable to those of PAV40. These results demonstrate the effectiveness of using the Thin Film PAV20 aging procedure in inducing extreme aging conditions in asphalt binders.

A detailed comparison between Thin Film PAV20 and PAV40 reveals that, at the high PG temperature, Thin Film PAV20 generally results in higher G* and lower δ than PAV40. At the intermediate PG temperatures, it produces values of G* and δ that are very close to those of PAV40. At the low PG temperatures, Thin Film PAV20 yields higher G* while maintaining a similar δ. Overall, Thin Film PAV20 and PAV40 exhibit comparable effects on the rheological properties of asphalt binders, with slight differences depending on the temperature.

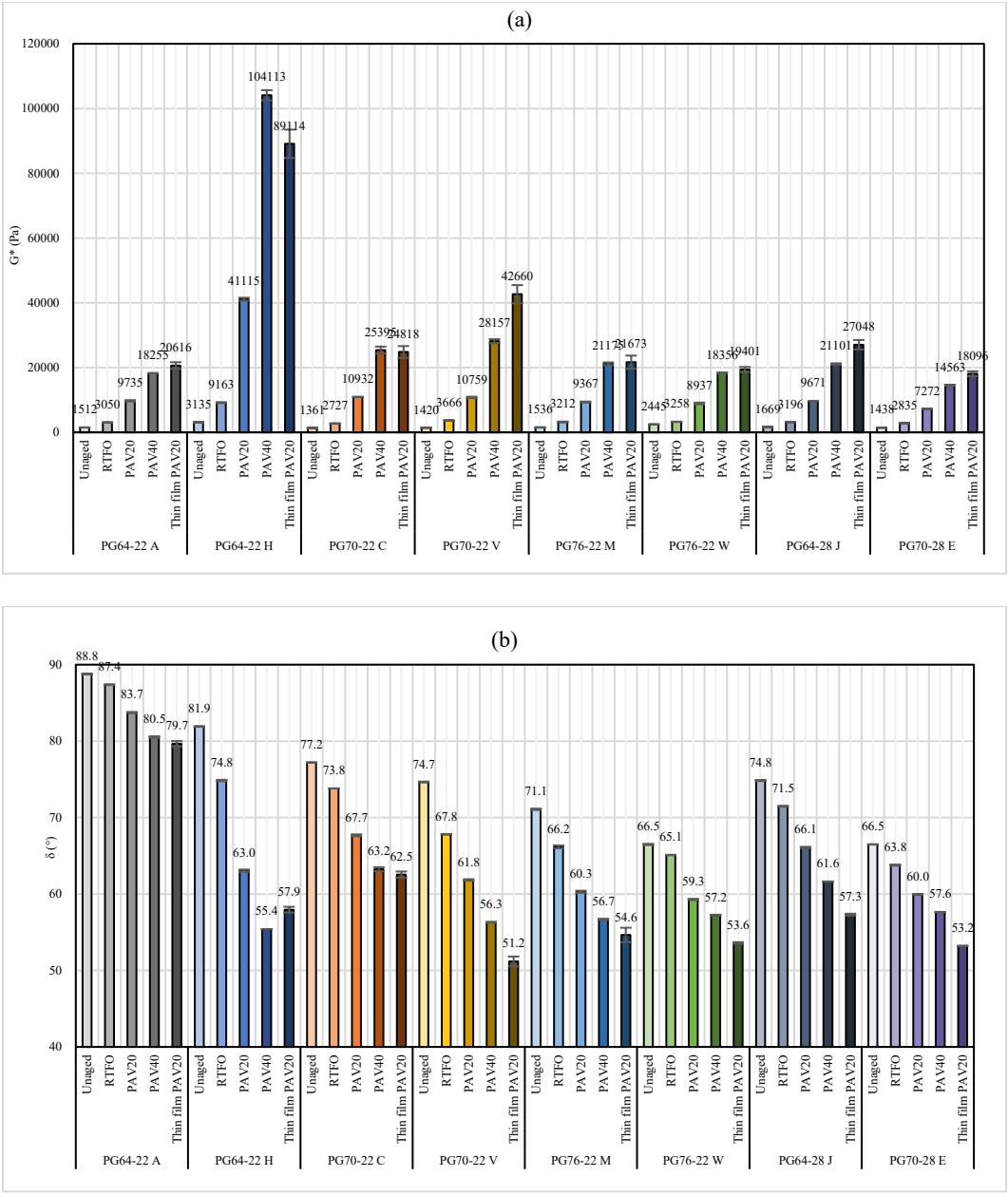


Figure 6.3 DSR results at high PG temperature: (a) G, (b) δ.*

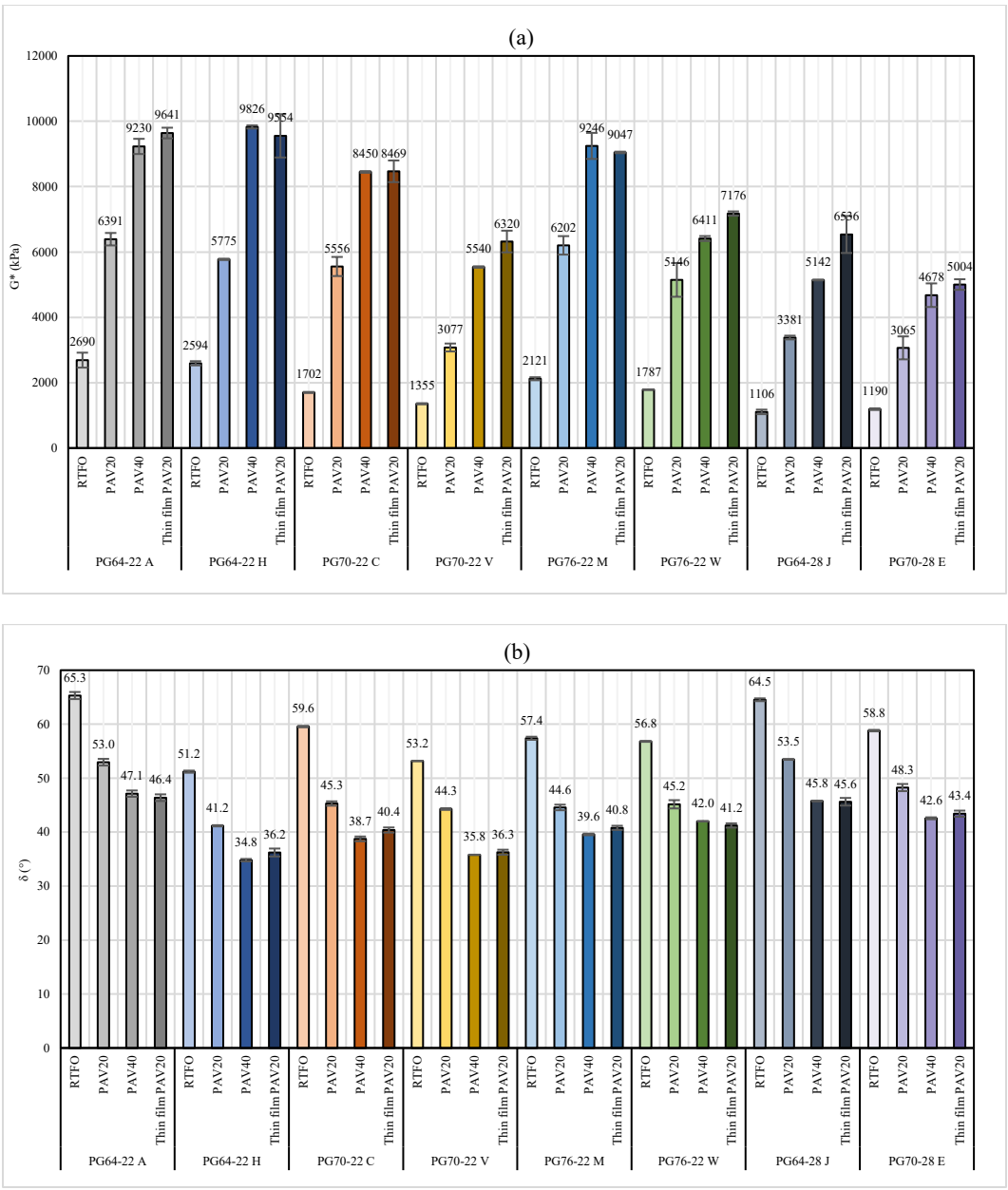


Figure 6.4 DSR results at intermediate PG temperature: (a) G, (b) δ.*

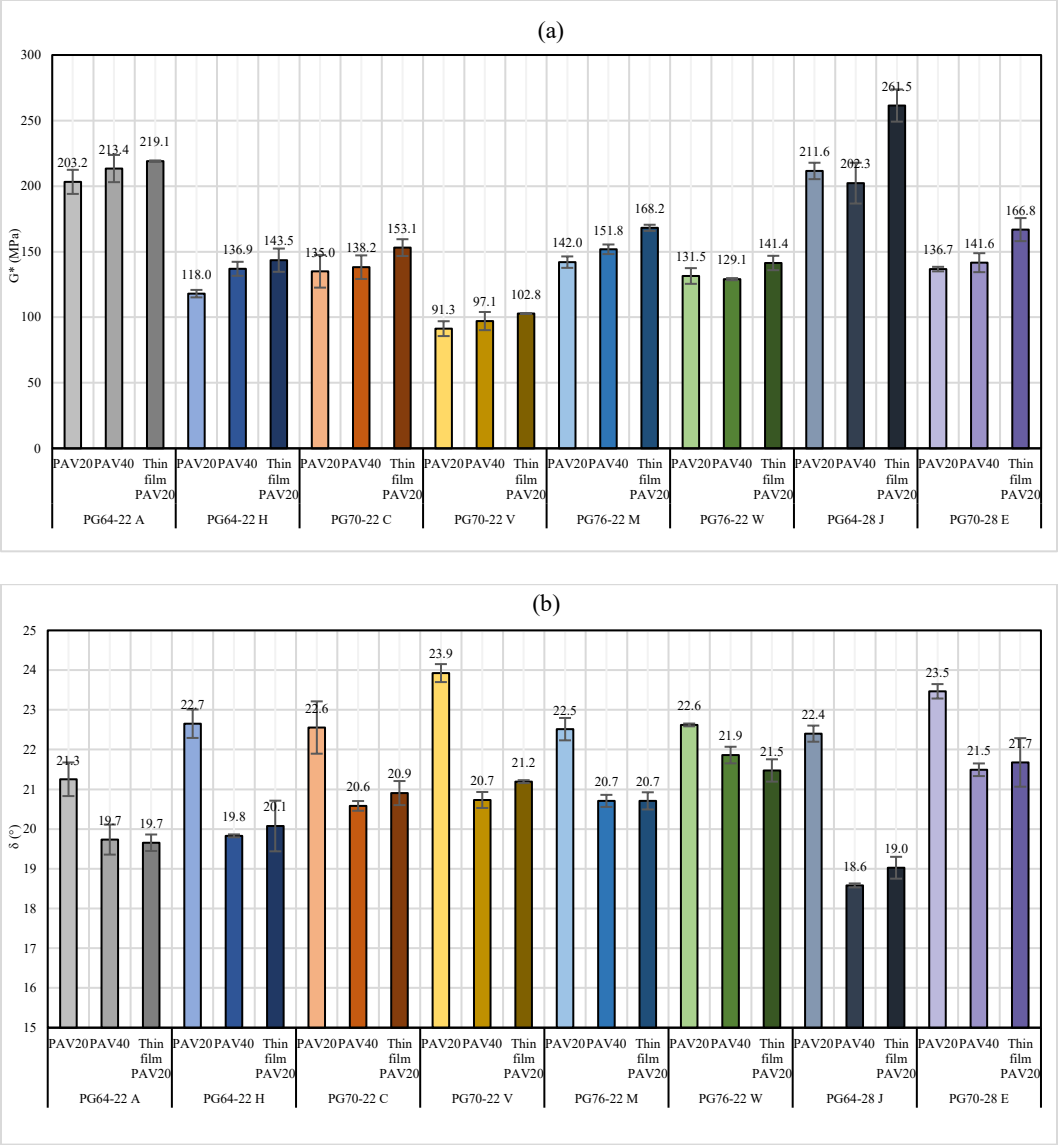


Figure 6.5 DSR results at low PG temperature: (a) G, (b) δ.*

To better illustrate the impact of long-term aging on binder performance, the intermediate temperature parameter G*sin(δ) of six asphalt binders at 25 °C was calculated. Although existing studies suggest that G*sin(δ) may not fully capture the actual cracking performance of asphalt binder and pavement, it can still serve as a rheology-based performance indicator for reference. A bump chart was constructed to visualize changes in performance ranking with increasing aging severity, as shown in Figure 6.6. It can be seen that the ranking of G*sin(δ) shows considerable variation from RTFO to PAV20, while the changes from PAV20 to PAV40 and Thin Film PAV20 are less pronounced. This indicates that long-term aging may have a significant impact on the performance of asphalt binders, highlighting the necessity of evaluating binder properties under extended aging conditions.

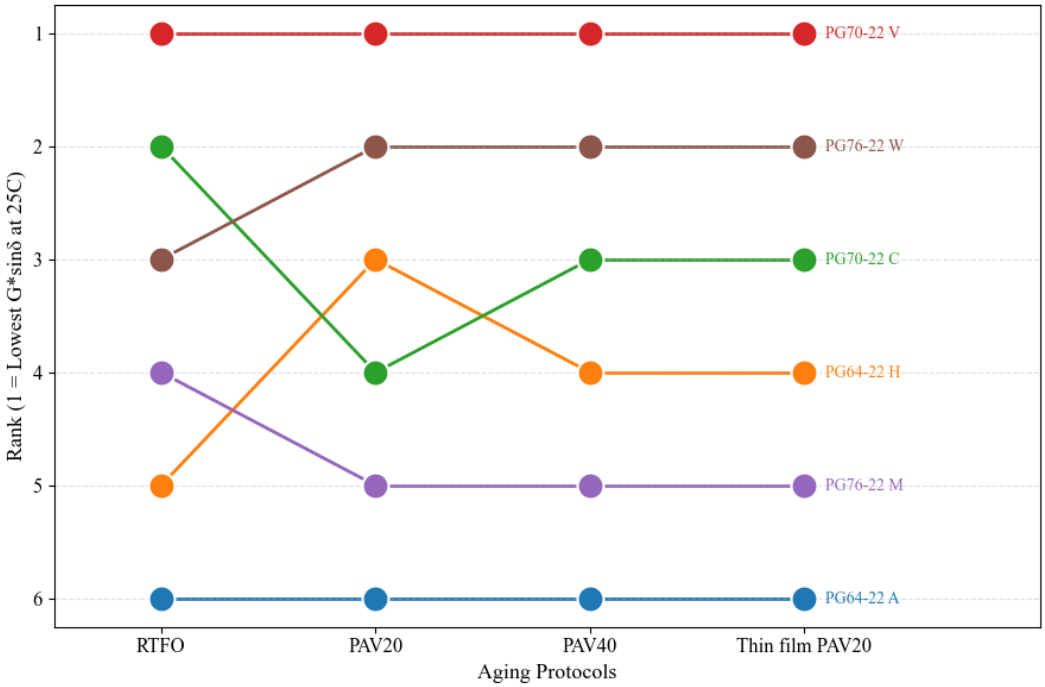
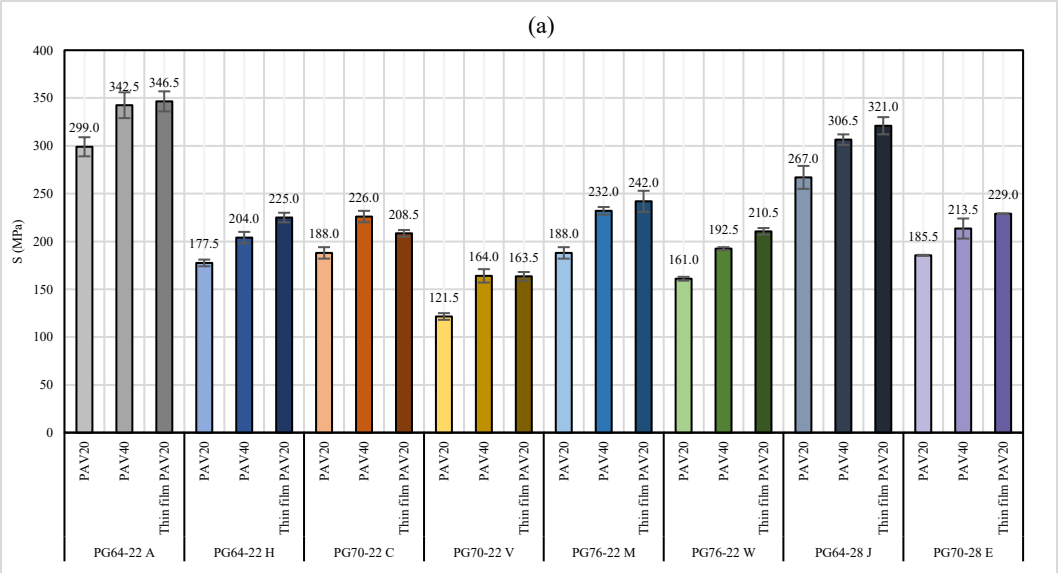


*Figure 6.6 Ranking of DSR G*sin(δ) at 25C Across Aging Protocols.*

6.1.4.2. BBR test results

Figure 6.7 presents the low-temperature rheological indices, stiffness and *m*-value, obtained from the BBR test at the low PG temperature. The results indicate that at low temperatures, Thin Film PAV20 leads to higher stiffness and lower *m*-value compared to PAV20. When compared to PAV40, it produces slightly higher stiffness and a similar *m*-value. These findings demonstrate that Thin Film PAV20 protocol can effectively accelerate binder aging with respect to low-temperature properties and induce an aging condition comparable to that of PAV40.



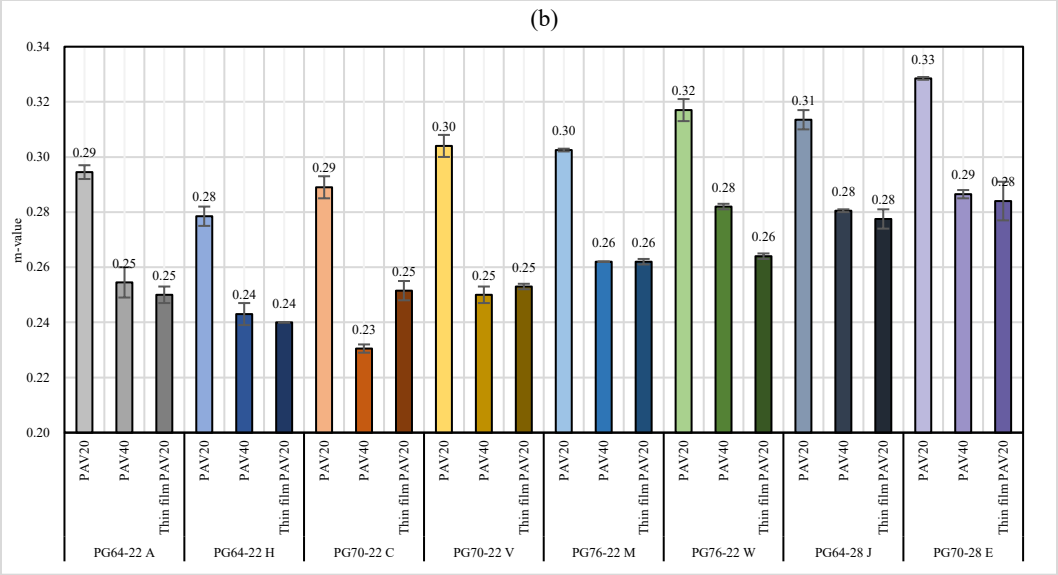


Figure 6.7 BBR results at low PG temperature: (a) Stiffness, (b) m-value.

Since the *m*-value reflects the binder's ability to relax stress at low temperatures and consistently serves as a critical parameter in determining the low-temperature PG grade, it is used as an indicator of low-temperature cracking performance. A bump chart was constructed based on the *m*-value to illustrate changes in the ranking of low-temperature cracking performance from PAV20 to PAV40 and Thin Film PAV20, as shown in Figure 6.8. It is observed that from PAV20 to PAV40 and Thin Film PAV20, the ranking of binder m-values changes significantly. This indicates that the degradation of low-temperature performance under long-term aging is inherently nonlinear, rather than a simple extension from PAV20. Therefore, it is necessary to adopt PAV40 and Thin Film PAV20 protocols which truly represent long-term aging when evaluating low-temperature performance.

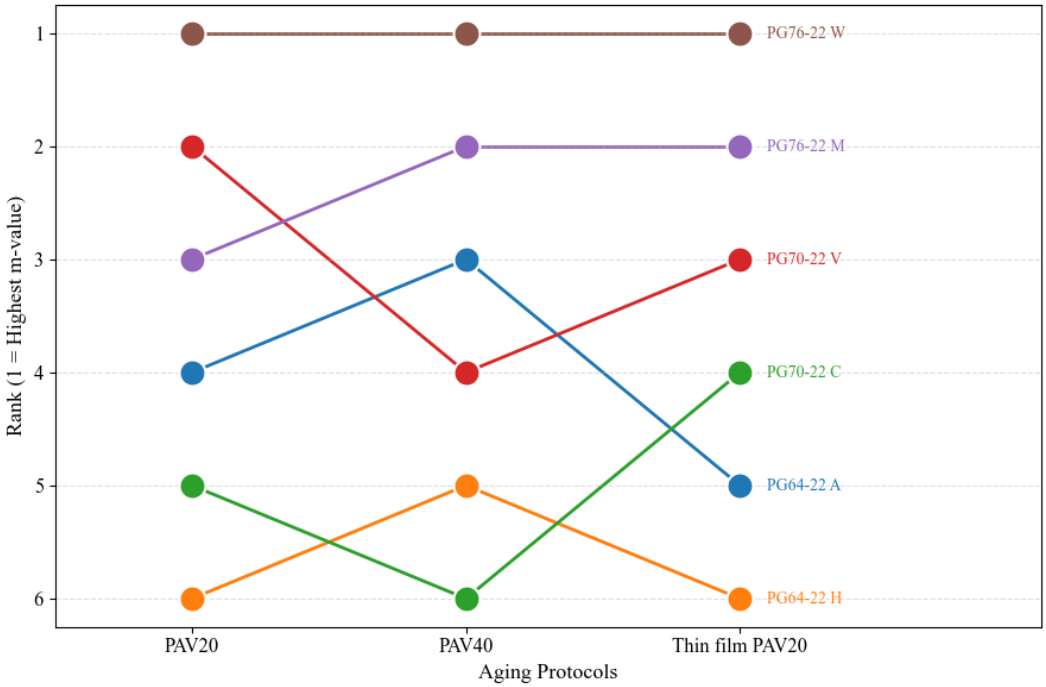


Figure 6.8 Ranking of BBR m-value at -12C Across Aging Protocols.

6.1.4.3. FTIR spectroscopy results

Figure 6.9 illustrates the variation trends of oxidation-related functional group indices in asphalt binders as determined by FTIR spectroscopy. Specifically, the carbonyl index (C=O) and sulfoxide index (S=O) were calculated to reflect the formation of oxidative products. The increase in these indices during the aging process is directly associated with the accumulation of the corresponding structural groups resulting from oxygen uptake in the asphalt bulk.

The results show that from RTFO to PAV40, both the C=O index and S=O index exhibit an overall increasing trend. Thin Film PAV20 yields higher oxidation indices than PAV20. A comparison between Thin Film PAV20 and PAV40 further reveals that the C=O index of Thin Film PAV20 is generally higher than that of PAV40, whereas the S=O index does not consistently follow this pattern. Additionally, Thin Film PAV20 exhibits greater variability in oxidation indices, which may be related to the uniformity of film thickness in PAV pans. Overall, Thin Film PAV20 and PAV40 produce comparable oxidation levels. These findings confirm the effectiveness of Thin Film PAV20 in accelerating long-term oxidative aging of asphalt binders from the perspective of chemical functional groups.

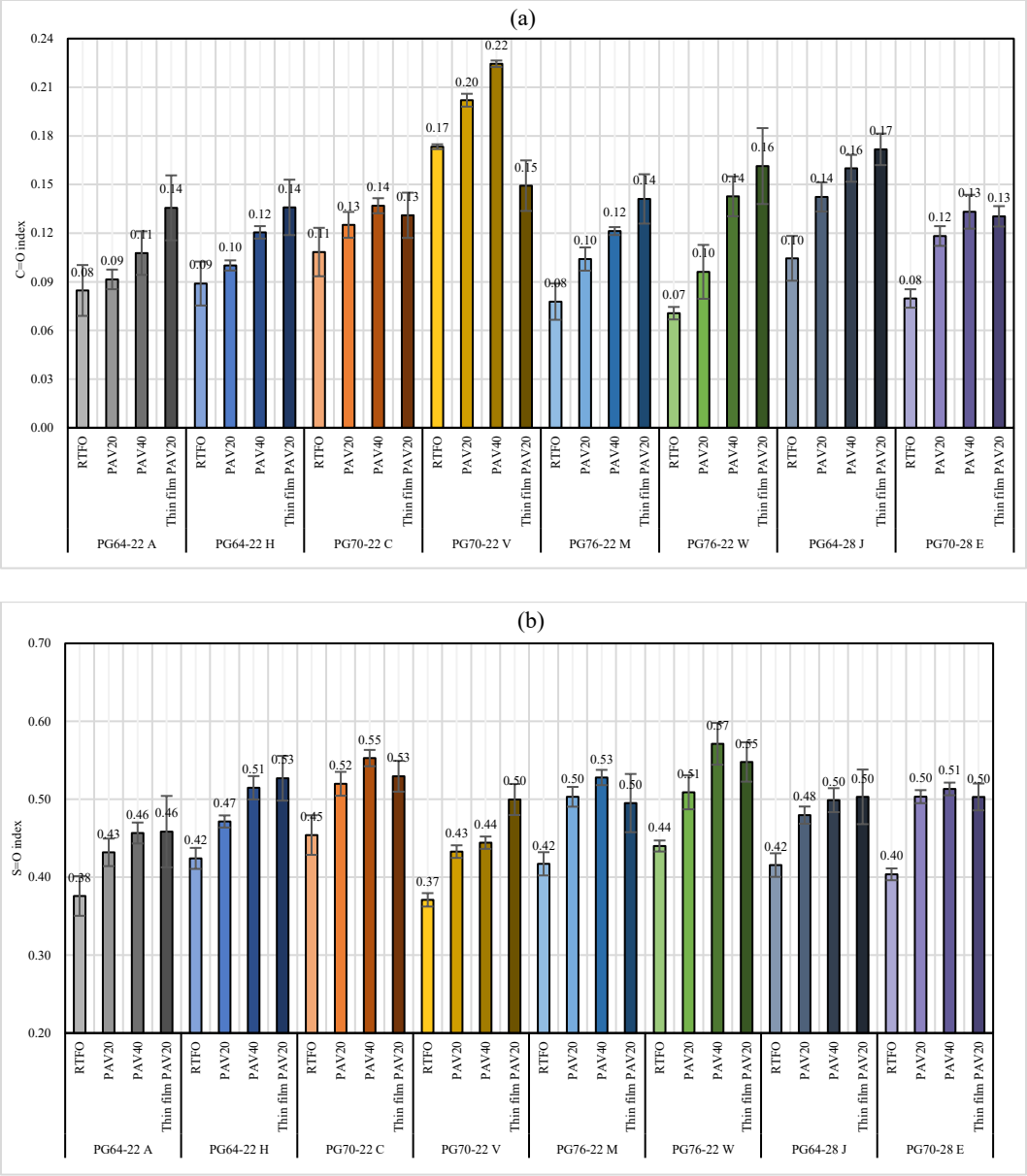


Figure 6.9 FTIR results: (a) C=O index, (b) S=O index.

6.1.4.4. Poker-chip test results

As shown in Figure 6.10, the poker chip test results indicate a progressive increase in tensile strength and a corresponding decrease in ductility from RTFO to PAV40, demonstrating a significant deterioration in cracking performance with aging. In comparison, Thin Film PAV20 exhibits notable differences from PAV20 in both parameters: its tensile strength consistently exceeds that of PAV40, while its ductility is similar to that of PAV40. This suggests that, from the perspective of cracking performance, Thin Film PAV20 can effectively simulate long-term aging.

An additional observation is that the ductility of modified binders declines sharply from RTFO to PAV20, whereas the two unmodified PG64-22 binders do not exhibit such deterioration. From PAV20 to PAV40 and Thin Film PAV20, ductility continues to decline significantly across all binders. This progression reduces the performance gap between modified and unmodified binders and leads to changes in the relative ranking of cracking performance among different binders.

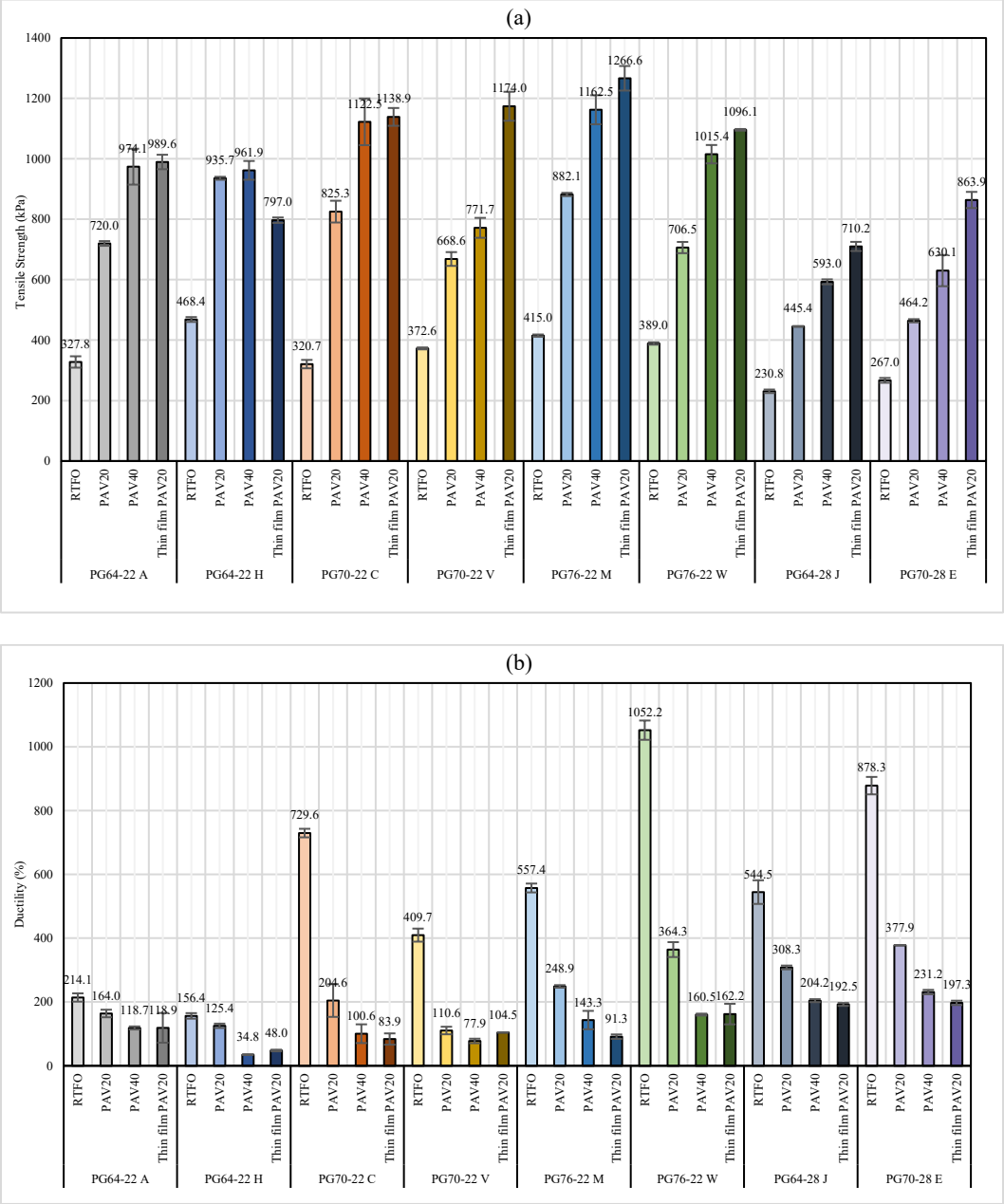


Figure 6.10 Poker-chip test results: (a) Tensile strength, (b) Ductility.

To further demonstrate the necessity of using PAV40 and Thin Film PAV20 as the long-term aging procedure, ductility was selected as the parameter for cracking evaluation, and a bump

chart was constructed as shown in Figure 6.11 to show the ranking of cracking performance at different aging protocols. It can be observed that from RTFO to PAV20, the ductility ranking of the eight binders undergoes significant changes, indicating that the effect of long-term aging on cracking performance is nonlinear and complex. Furthermore, from PAV20 to PAV40 and Thin Film PAV20, the performance ranking changes again. This suggests that the effects of extended aging are not simply a continuation of the trends observed under PAV20, but rather involve further differentiation and reordering of cracking performance across binders. Besides that, it is shown that the cracking performance rankings under PAV40 and Thin Film PAV20 show minimal differences, which indirectly supports the conclusion that both aging protocols induce a similar level of aging. As such, evaluating long-term cracking behavior requires aging protocols beyond PAV20 to capture this evolving material response, which emphasizes the importance and necessity of using PAV40 and Thin Film PAV20 protocols.

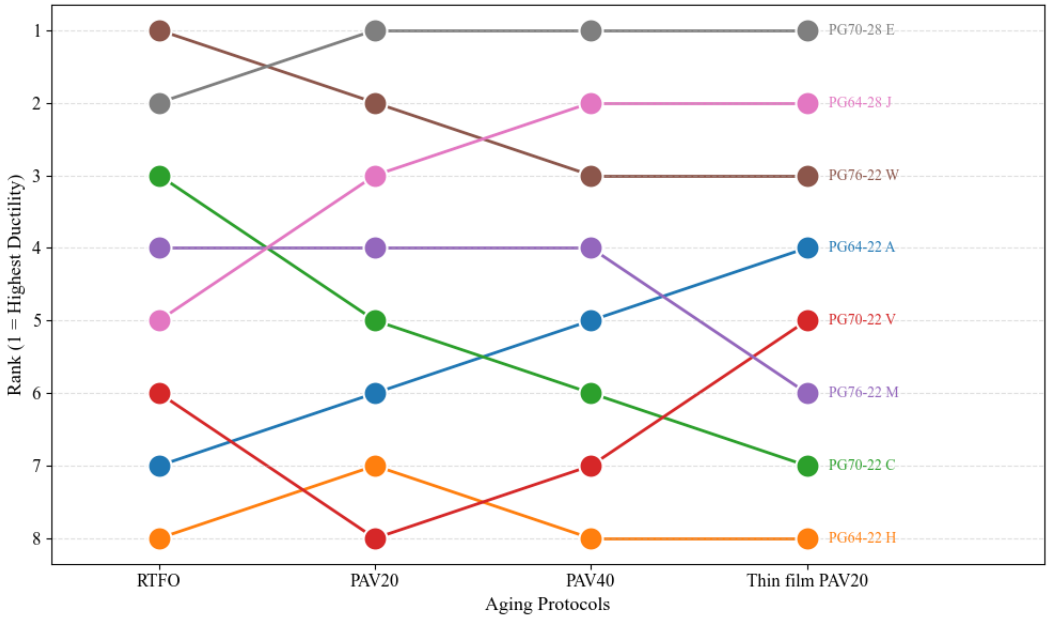


Figure 6.11 Ranking of Poker-chip ductility at 25C Across Aging Protocols.

6.1.5. Summary

In this part of the study, the accelerated long-term aging protocol for asphalt binder was presented. DSR and BBR tests were employed to compare the rheological properties of asphalt binders under different long-term aging protocols at high, intermediate, and low temperatures. FTIR spectroscopy was used to evaluate the influence of each aging protocol on oxidative aging from the perspective of chemical functional groups. The poker chip test was conducted to investigate binder cracking performance at intermediate temperatures. The conclusions are as follows:

1. In terms of all the rheological, chemical, and cracking performance, the Thin Film PAV20 aging protocol induces significantly more advanced aging conditions in asphalt binders compared to traditional PAV20, while exhibiting aging effects comparable to those of PAV40.

2. By comparing the ranking of cracking performance of asphalt binders under different aging conditions, it is evident that substantial changes occur from RTFO to PAV20, highlighting the necessity of employing long-term aging protocols to evaluate cracking resistance. Furthermore, the transition from PAV20 to PAV40 and Thin Film PAV20 results in additional noticeable changes, indicating that the effects of extended aging are not simply a continuation of the trends observed under PAV20, but rather involve further differentiation and reordering of cracking performance across binders. This confirms the importance of using PAV40 and Thin Film PAV20 as the representative long-term aging protocols.

6.2. Evaluating the Accelerated Long-term Aging Protocol for Asphalt Mixture

6.2.1. Goal

Although asphalt binder properties are important, it is the asphalt mixture that ultimately influences field performance of asphalt pavements. The goal of this exercise is to track the accelerated aging protocol for loose mixes and explore the influence of long-term aging on the cracking performance of asphalt mixture.

6.2.2. Evaluation

6.2.2.1. Aging protocol

Four aging condition levels for asphalt mixture, which rely on increasing the aging temperature instead of aging time, were applied to age each loose mix before preparing mixture specimens for cracking tests, as described below:

1. Standard short-term aging in Texas: 2-hr. at compaction temperature.

2. Long-term aging [99]:

    1) 20-hr. at 100 °C

    2) 20-hr. at 110 °C

    3) 20-hr. at 120 °C

Three aging condition levels for asphalt binder were applied to each binder to fabricate loose mix:

1. Standard short-term aging: RTFO.
2. Long-term aging:
   1) PAV20
   2) PAV40

6.2.2.2. Evaluation procedure and test method

Step 1: Three binders with different performance grading and different suppliers were selected for laboratory binder aging (RTFO, PAV20 and PAV40). The same binders were also used to prepare loose mixes using the same aggregate and gradation, which were subjected to mixture aging (short-term aging and three long-term aging: 20hr. at 100°C, 20hr. at 110°C, and 20hr. at 120°C).

Step 2: The asphalt binder was extracted and recovered for each loose mix after long-term aging.

Step 3: DSR and poker-chip tests were used to compare the aging conditions between laboratory-aged binders and extracted and recovered binders.

Step 4: Three more groups of asphalt mixtures with different binders, RAP contents, and aggregate types were prepared in the lab and subjected to short-term and long-term aging conditions.

Step 5: IDEAL Cracking Test (IDEAL-CT) and Overlay Test (OT) were conducted for each group of mixtures to explore the cracking performance after long-term aging.

6.2.2.2.1. Binder extraction and recovery

A centrifuge extractor was used with toluene as the solvent to recover the asphalt binder in a solvent from a sample of the loose asphalt mixture. The rotary evaporator method was used to recover the asphalt binder from the solvent following the method described in AASHTO TP2 (1999).

6.2.2.2.2. DSR test

The DSR was used for performance testing and to compare the rheological properties between laboratory-aged binders and extracted and recovered binders. A temperature sweep test was used to evaluate binder properties at high and intermediate PG temperatures. The 8 mm DSR test (proposed replacement for BBR) was used to test binders at low PG temperature (not applicable for RTFO binder). Two replicates were used for each measurement. The parameter G* and δ from the last five cycles was averaged. Both parameters were used as the rheological indicator.

The DSR test procedures are the same shown in Section 6.1.3.2.1 using the corresponding testing temperatures for different binders as shown in Table 6.1.

6.2.2.2.3. Poker-chip test

Poker-chip test was used to compare the cracking performance between laboratory-aged binders and extracted and recovered binders. The strength and ductility parameters obtained from the poker-chip test were used for this comparison. The sample preparation procedures and detailed experimental protocols are described in the preceding sections. Two replicates were used for each sample.

6.2.2.2.4. IDEAL-CT: Monotonic Cracking Test

The IDEAL-CT (Tex-250-F) is a monotonic cracking test for determining the cracking tolerance index (CT_{index}) of compacted asphalt mixtures. A higher CT_{index} indicates better resistance to cracking in the asphalt mixture. The test is cast with a loading rate of 50 mm/min at 25°C. For each mix scenario, five IDEAL-CT specimens were molded using a Superpave gyratory compactor to achieve a consistent air void level of 7 ± 0.5%. These specimens have a diameter of 150 mm and a height of 62 mm. Prior to testing, each specimen was conditioned in a temperature chamber for two hours at 25°C. Five replicates for each set were averaged. The determination of the CT_{index} involved analyzing the load-displacement curves recorded during the testing process, as depicted in Figure 6.12, and applying the equation below for the calculation.

$$CTindex = \frac{t}{62} \times \frac{l_{75}}{D} \times \frac{G_f}{|m_{75}|} \times 10^6$$

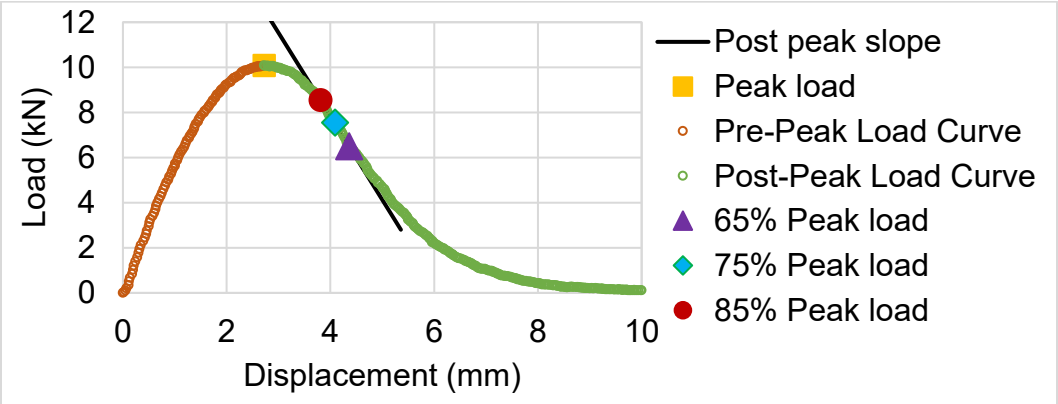


Figure 6.12 IDEAL-CT Load-Displacement Curve.

6.2.2.2.5. Overlay Test: Cyclic Cracking Test

The Overlay Test (OT) (Tex-248-F) is a cyclic cracking test for evaluating the susceptibility of asphalt mixtures to fatigue or reflective cracking. The key parts of the apparatus consist of two steel plates, one fixed and the other which moves horizontally to simulate the opening and closing of joints or cracks in old pavements beneath an overlay. The OT is conducted in a cyclic triangle displacement-controlled mode with a maximum opening of 0.025 inch at room temperature of 25°C and at a loading frequency of 0.1 Hz (ten seconds per cycle). The OT specimen has a dimension of 6-inch-long by 3-inch-wide by 1.5-inch-high cut from Superpave gyratory compacted samples.

To estimate the number of cycles to failure, the 93% load reduction method was applied. This involves plotting the peak load for each cycle against the number of cycles, as illustrated in Figure 6.13. The Crack Progression Rate (CPR) is determined by fitting a power equation to the peak load versus the number of cycles curve, as depicted in Figure 6.13. This index serves as an indicator of the mix's cracking resistance, with lower values indicating superior performance in terms of crack resistance. In addition, critical fracture energy can be obtained by calculating the area under the load vs. displacement curve as shown in Figure 6.14.

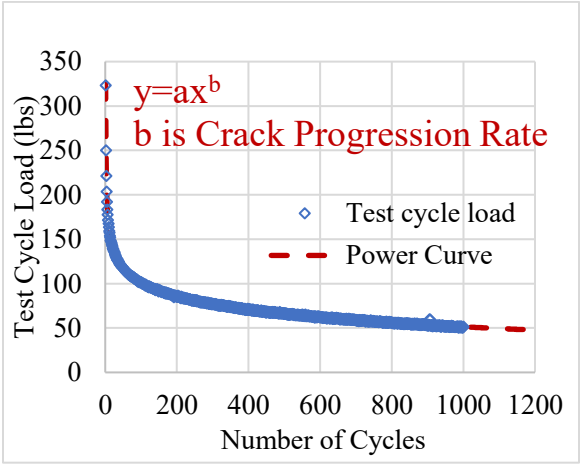


Figure 6.13 Maximum load versus cycles.

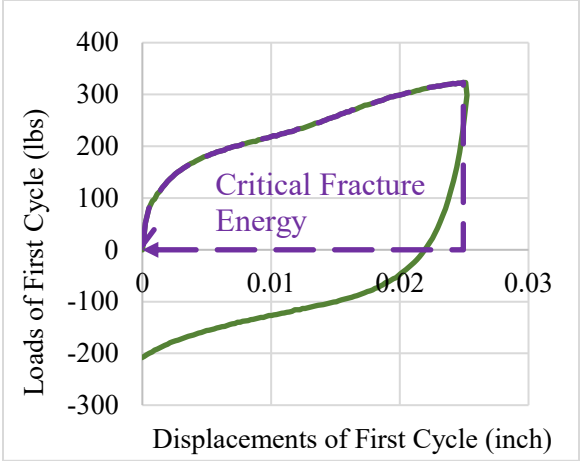


Figure 6.14 Hysteresis loop.

6.2.2.3. Materials

Three binders and corresponding loose mixes (limestone, SP-C) were used to compare the aging conditions between laboratory-aged binders and extracted and recovered binders:

• PG64-22 A

• PG70-22 C

• PG76-22 M

Three groups of asphalt mixtures as shown in Table 6.2 were further prepared and aged to study the effect of long-term aging on the cracking performance of asphalt mixture. The two most used mixtures in Texas are 12.5 mm Superpave-C and 9.5 mm Superpave-D. Two aggregates (crushed limestone and gravel) and associated gradations (SP-C and SP-D) are included in the study to ensure that the research findings are meaningful and applicable. The gradation details are shown in Figure 6.15.

Table 6.2 Summary of mixture testing plan

Binder	RAP Content (%)	Aggregate	Aging Conditions	Cracking Tests
PG 64-22 A	20	Gravel SP-D	2hrs at compact temperature	IDEAL cracking test (IDEAL-CT) and Overlay test (OT)
			20h@100C	
			20h@110C	
			20h@120C	
PG 70-22 C with P25	0	Limestone SP-C	2hrs at compact temperature	
			20h@100C	
			20h@110C	

			20h@120C	
PG 76-22 M	0	Limestone SP-C	2hrs at compact temperature	
			20h@100C	
			20h@110C	
			20h@120C	

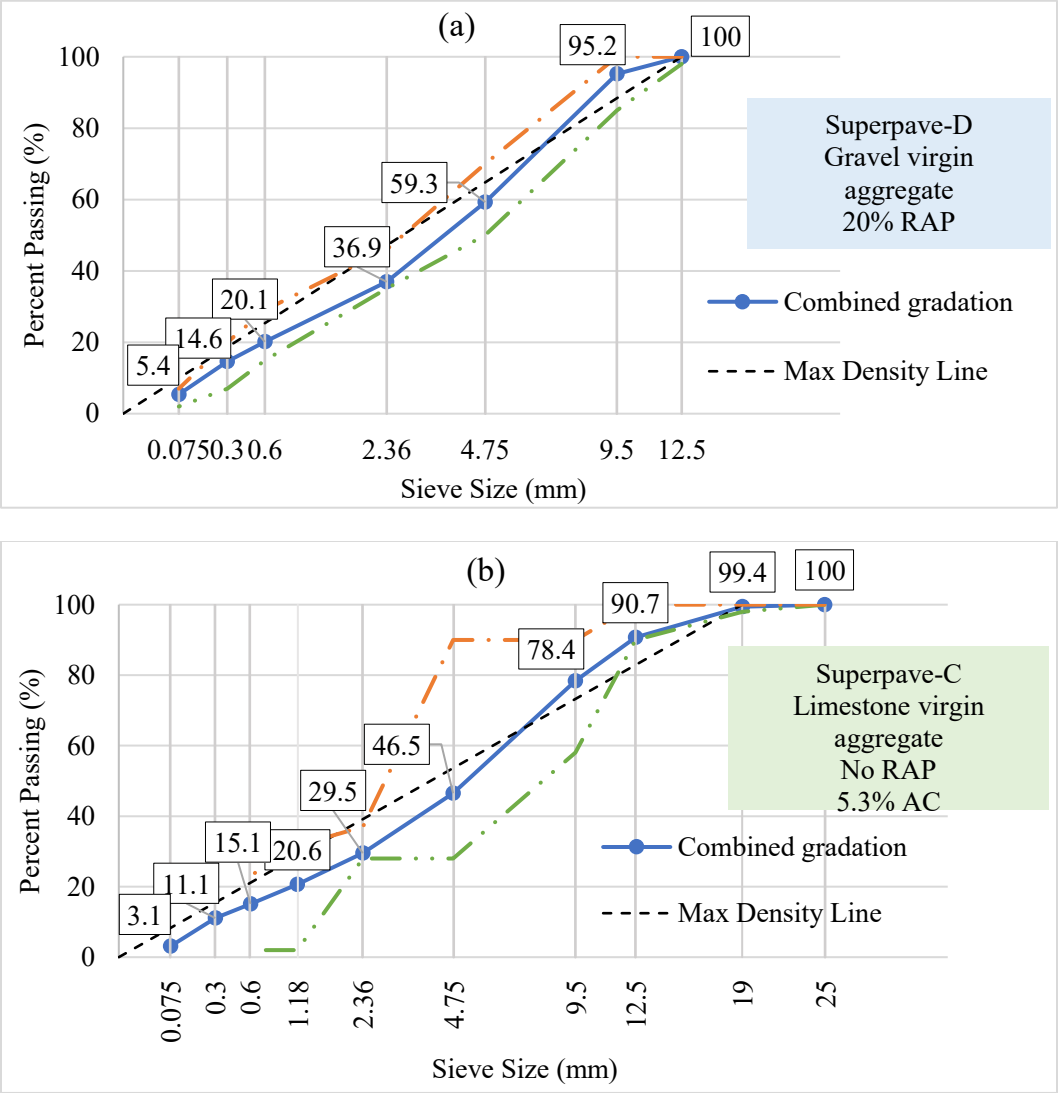


Figure 6.15 Mixture gradations: (a) Superpave-D with gravel; (b) Superpave-C with limestone.

6.2.3. Results and Discussion

6.2.3.1. Comparison of the degree of aging

Figure 6.16 to Figure 6.18 present the raw DSR data for three binders tested at high-, intermediate-, and low-PG temperatures. The RTFO through PAV40 represent laboratory-aged

binders, while Extraction 20hr. at 100°C, Extraction 20hr. at 110°C, and Extraction 20hr. at 120°C refer to binders extracted and recovered from aged loose mixes.

In general, it is evident that at all PG temperatures, extending PAV aging duration and increasing the aging temperature for loose mixes significantly increases the G* and reduces the δ, which aligns with general expectations. It confirms that increasing the aging temperature of asphalt mixture can effectively accelerate the aging condition of asphalt binder.

In addition, when compared to PAV20, it is observed that the G* values of Extraction 20hr. at 100°C are closely aligned with those of PAV20 across all three temperatures, while the δ of Extraction 20hr. at 110°C appear to be similar to those of PAV20 at intermediate and low temperatures. In comparison with PAV40, the rheological parameters of Extraction 20hr. at 120°C are found to be comparable. A detailed comparison is provided in the following section.

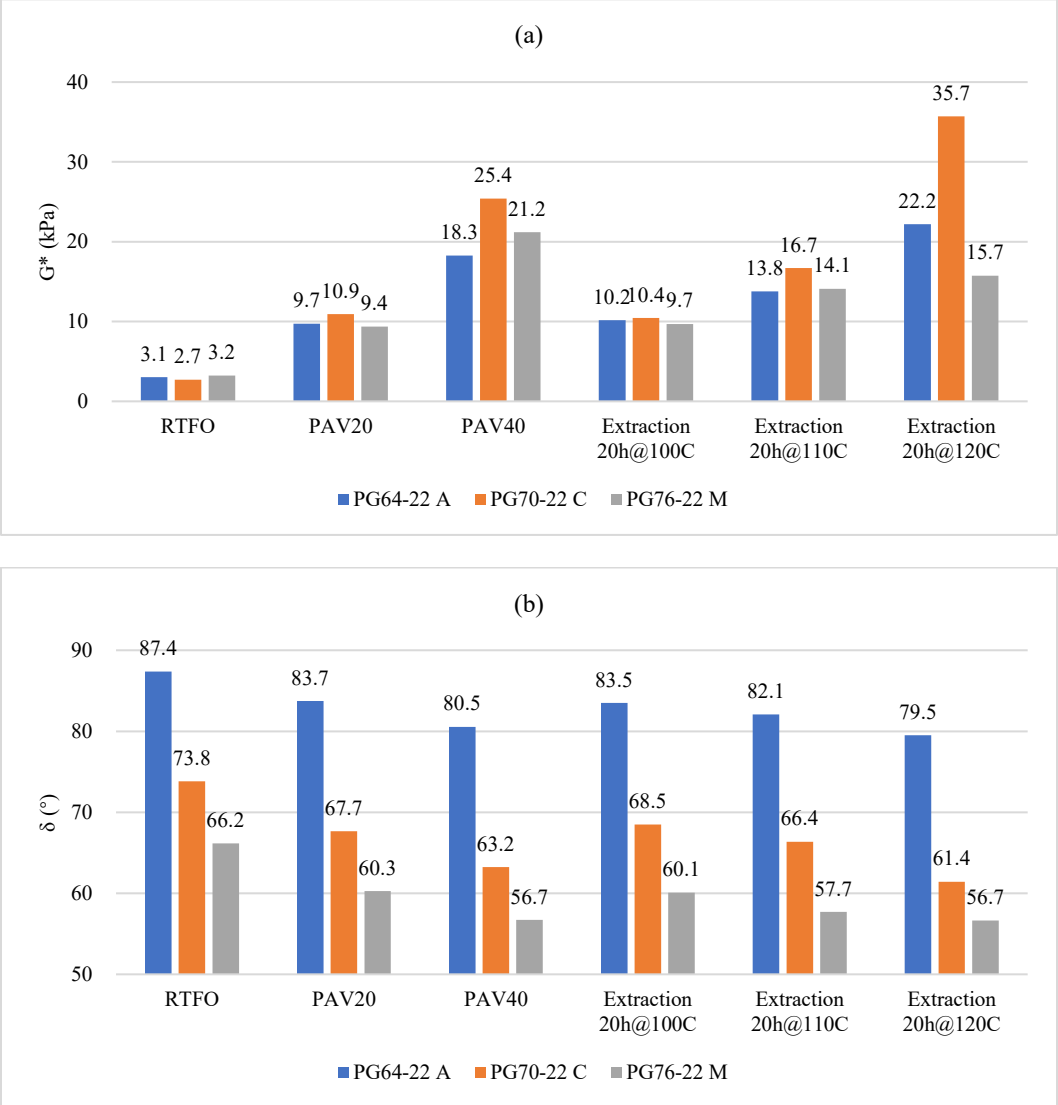


Figure 6.16 DSR results at high PG temperature: (a) G, (b) δ.*

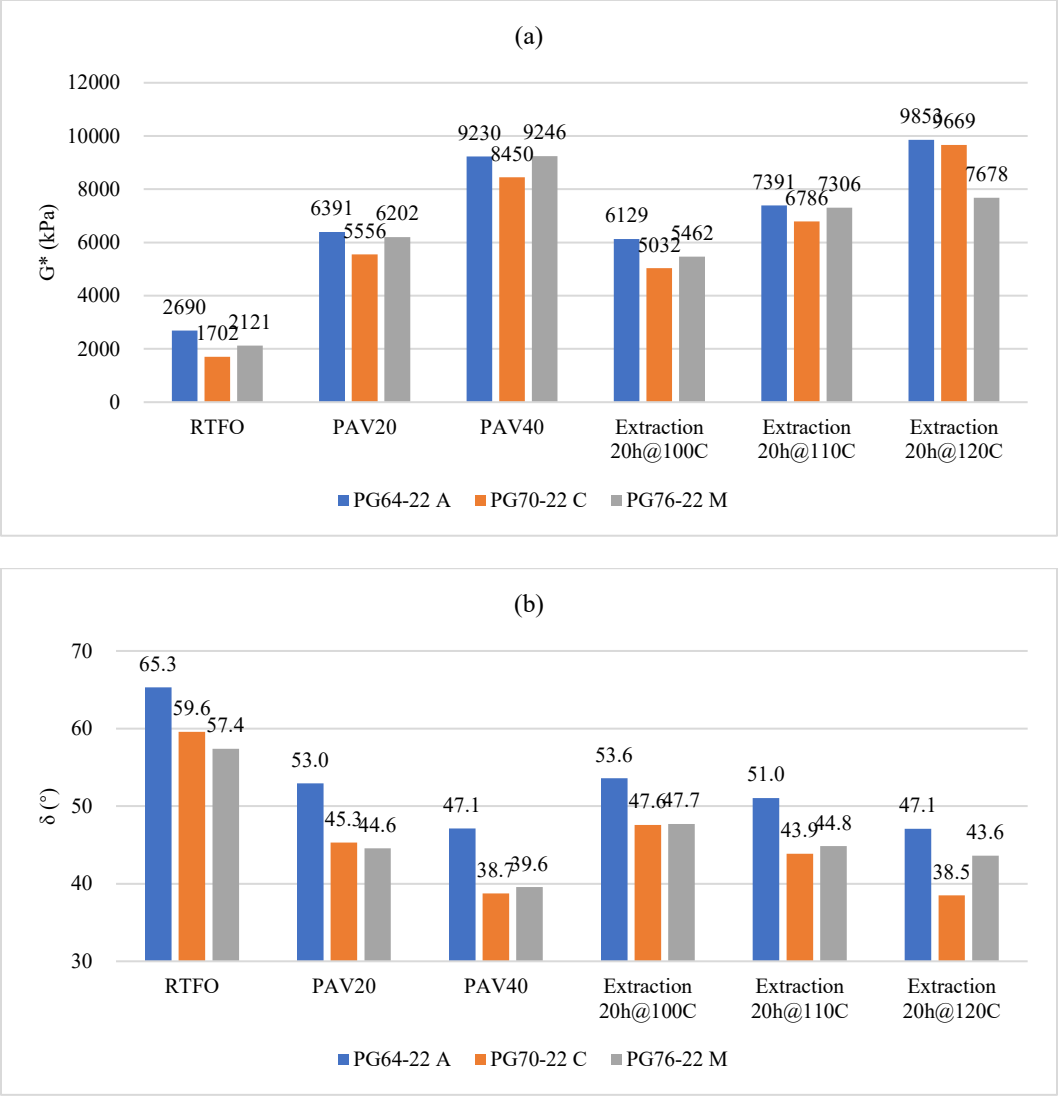


Figure 6.17 DSR results at intermediate PG temperature: (a) G, (b) δ.*

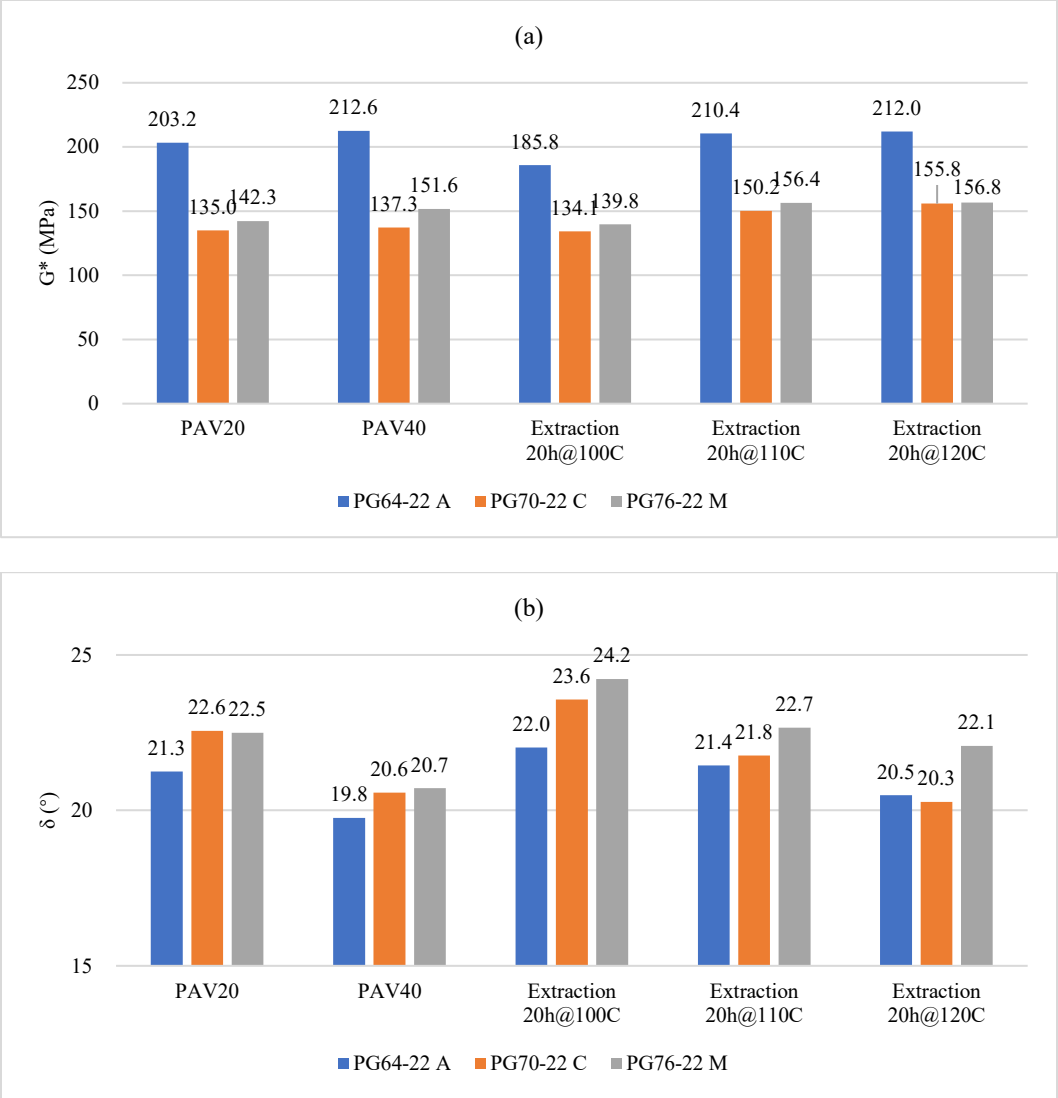


Figure 6.18 DSR results at low PG temperature: (a) G, (b) δ.*

Figure 6.19 shows the results of the poker chip test. For laboratory-aged binders, aging from RTFO to PAV40 leads to a consistent increase in tensile strength and a corresponding decrease in ductility. For the extracted and recovered binders, an overall trend is observed in which higher aging temperatures result in increased tensile strength and reduced ductility. This is consistent with the known effects of aging on the cracking performance of asphalt binders. Exceptions include the highest tensile strength of PG70-22 C at Extraction 20hr. at 110°C, and the lowest ductility of PG64-22 A at Extraction 20hr. at 100°C. This trend indeed deviates from typical expectations. And PG70-22 and PG76-22 show little change in ductility from Extraction 20hr. at 110°C to 120°C, possibly due to the nonlinear effect of aging temperature on the cracking performance of modified binders. However, due to the limited amount of extracted and recovered binder, additional replicate tests could not be conducted. Further validation may be required in future.

As to the comparison, Extraction 20hr. at 100°C and PAV20 exhibit similar cracking performance, while Extraction 20hr. at 120°C shows comparable cracking parameters to PAV40. A detailed comparison is provided in the following section.

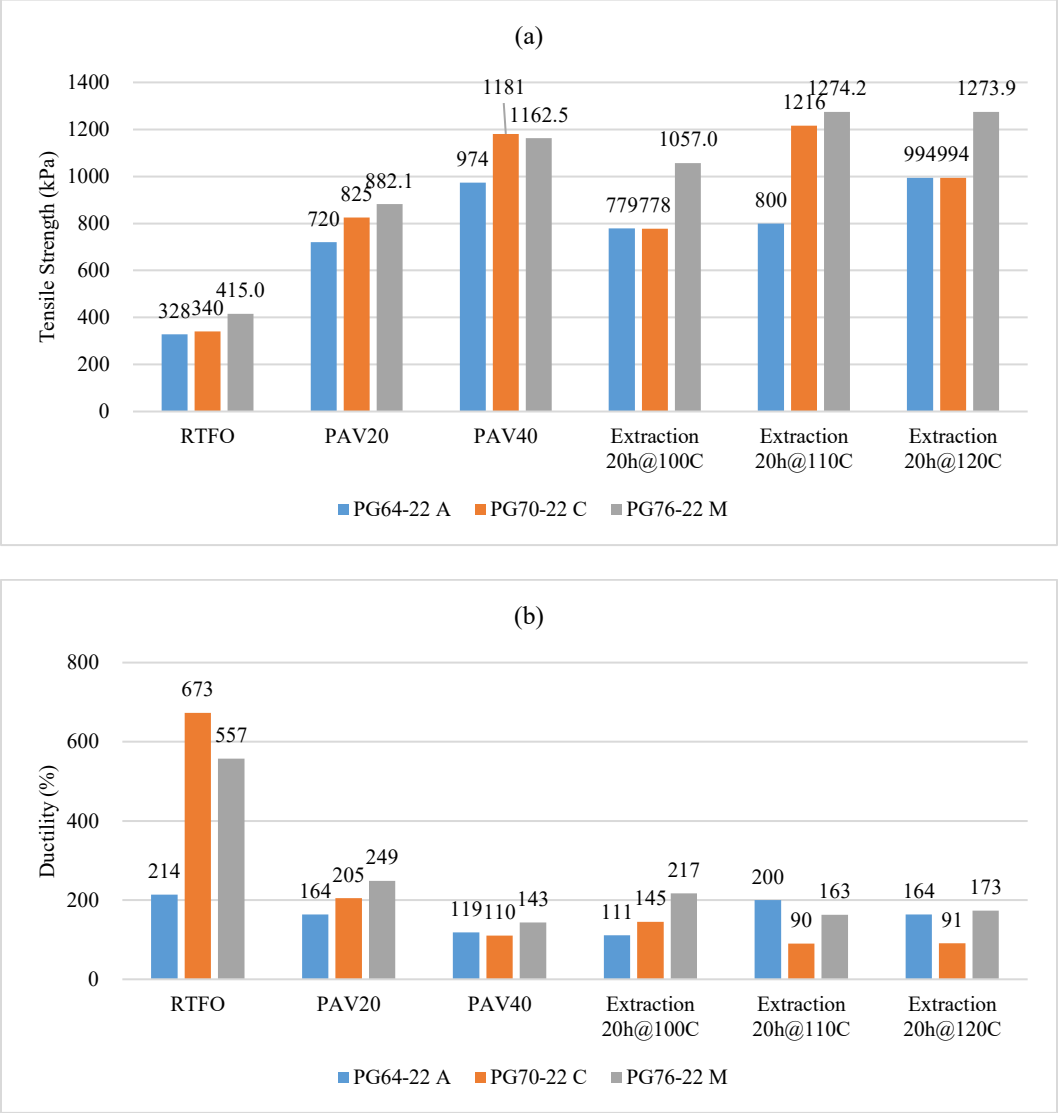


Figure 6.19 Poker-chip test results: (a) Tensile Strength, (b) Ductility.

To better compare the impact of different aging protocols on the performance of asphalt binders, all DSR and Poker-chip test results are expressed as the percentage difference between extracted and recovered binders and laboratory-aged binders. A new index, Difference of Aging (DOA), has been proposed to identify which long-term aging procedure of mixture (20hr. at 100°C, 20hr. at 110°C, 20hr. at 120°C) is mostly close to long-term aging procedure of binder (PAV20 or PAV40).

Difference of Aging (DOA) compared to PAV20 (%) is calculated as shown below:

$$DOA_{20} = \frac{X_y - X_{PAV20}}{X_{PAV20}} \times 100$$

Difference of Aging (DOA) compared to PAV40 (%) is calculated as shown below:

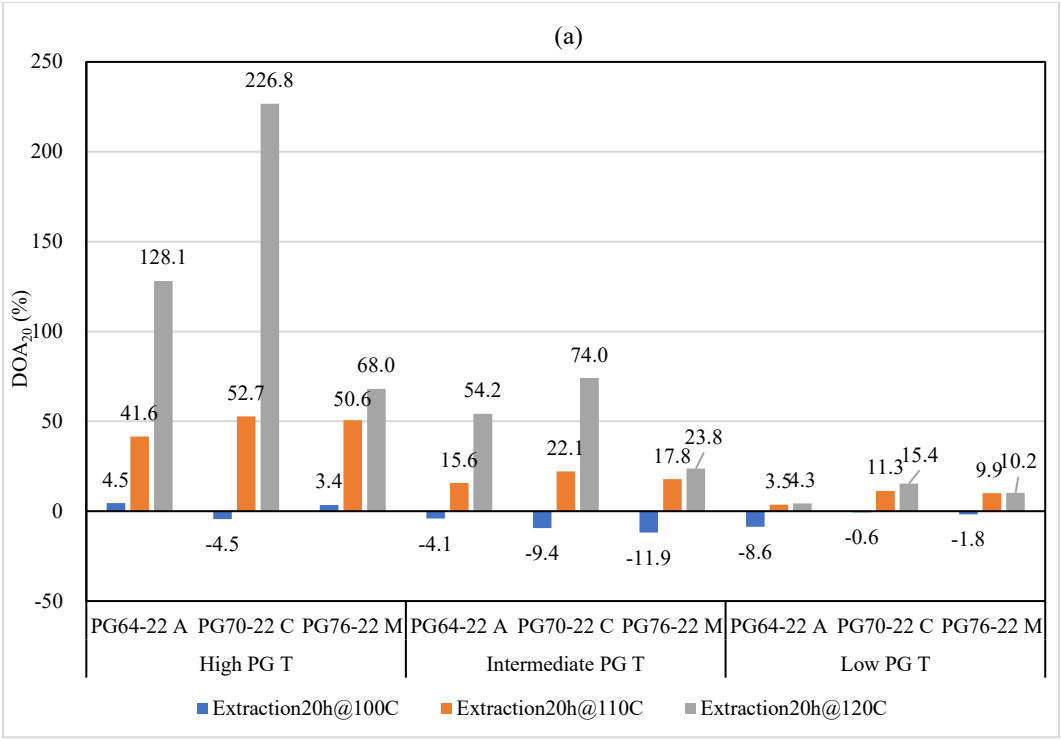
$$DOA_{40} = \frac{X_y - X_{PAV40}}{X_{PAV40}} \times 100$$

X represents any testing metric under specific aging condition and now are designated in Poker-chip parameters at intermediate temperature and DSR parameters at high, intermediate, and low PG temperature.

y represents the aging procedure of mixture (20hr. at 100°C, 20hr. at 110°C, and 20hr. at 120°C).

For example, a positive value of DOA_{20} indicates that the selected parameter under a given aging condition is aged more compared to PAV20 aging. The closer the DOA_{20} value is to zero, the more similar the parameter is between the two aging protocols.

Figure 6.20 and Figure 6.21 present the DOA_{20} values for DSR and poker chip test parameters. Overall, Extraction 20hr. at 100°C exhibits smaller DOA_{20} values compared to other conditions, indicating a similar level of aging to PAV20. Specifically, for the DSR parameters, G* at high, intermediate, and low temperatures under Extraction 20hr. at 100°C are very close to those under PAV20, suggesting comparable binder stiffness. In contrast δ shows a different trend: at high temperatures, Extraction 20hr. at 100°C aligns more closely with PAV20, whereas at intermediate and low temperatures, Extraction 20hr. at 110°C provides a better match. This suggests that the influence of aging temperature of loose mixes on binder viscoelastic properties may vary slightly across temperature range. For the poker chip test parameters, except for the ductility of PG64-22 A, all other cases show lower DOA_{20} values for Extraction 20hr. at 100°C. This indicates that, in terms of binder cracking performance, Extraction 20hr. at 100°C can produce a comparable aging effect to that of PAV20.



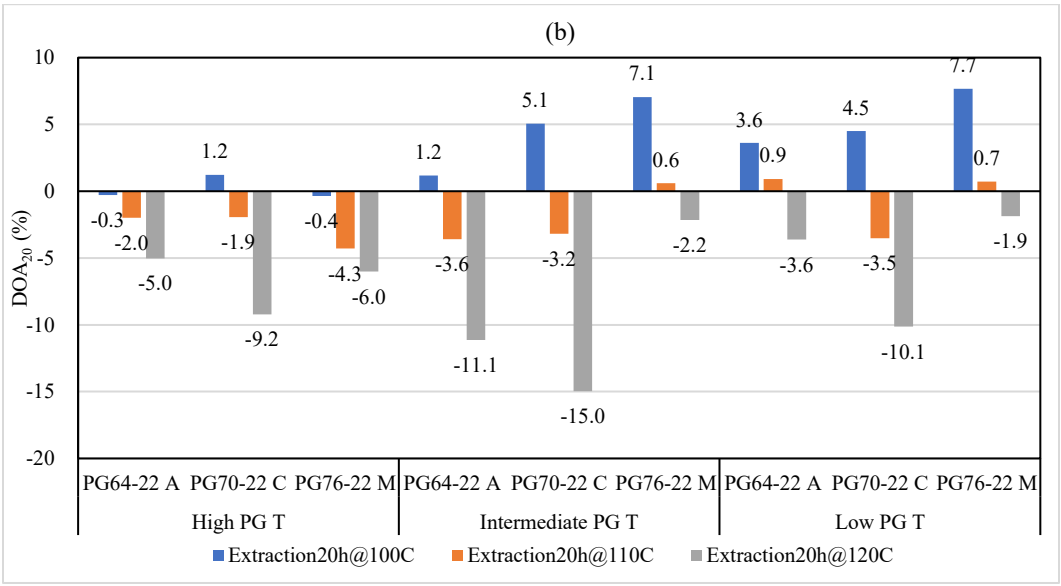


Figure 6.20 DOA_{20} for DSR parameters at three PG temperatures: (a) G, (b) δ.*

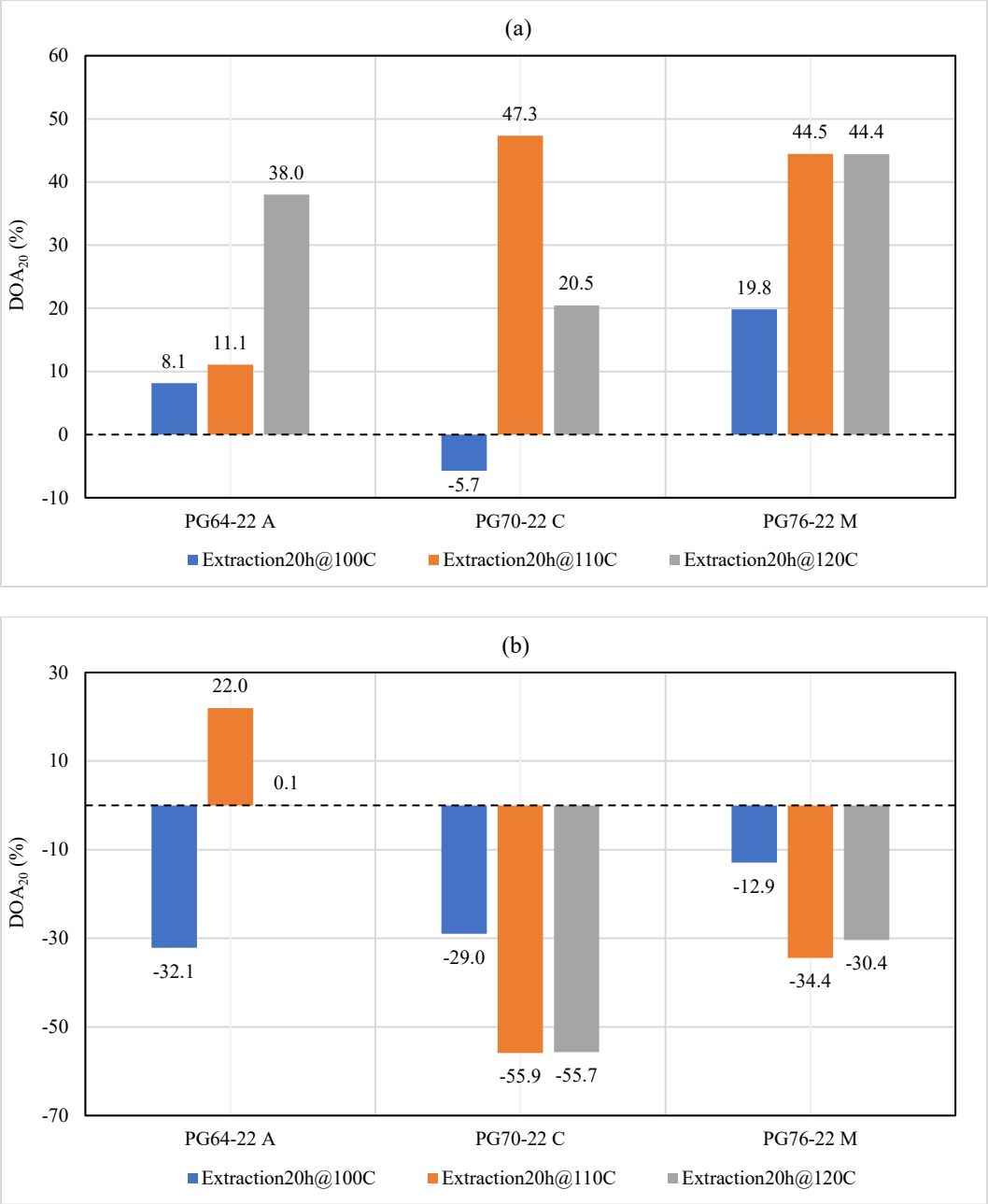


Figure 6.21 DOA_{20} for Poker-chip test parameters: (a) Peak stress, (b) Ductility.

Figure 6.22 and Figure 6.23 present the DOA_{40} values for the DSR and poker chip test parameters. Overall, the indicators under Extraction 20hr. at 120°C exhibit smaller DOA_{40} values, suggesting an aging level comparable to that of PAV40. Specifically, for the DSR parameters, both G* and δ under Extraction 20hr. at 120°C closely match those of PAV40 across high, intermediate, and low temperatures, indicating similar rheological behavior. For the poker chip test parameters, the influence of increased aging temperature on cracking performance does not always follow a consistent or expected trend. While Extraction 20hr. at 110°C and Extraction 20hr. at 120°C generally result in more severe cracking than Extraction 20hr. at 100°C, the

DOA_{40} values between Extraction 20hr. at 110°C and 120°C show no clear dominance, varying from case to case.

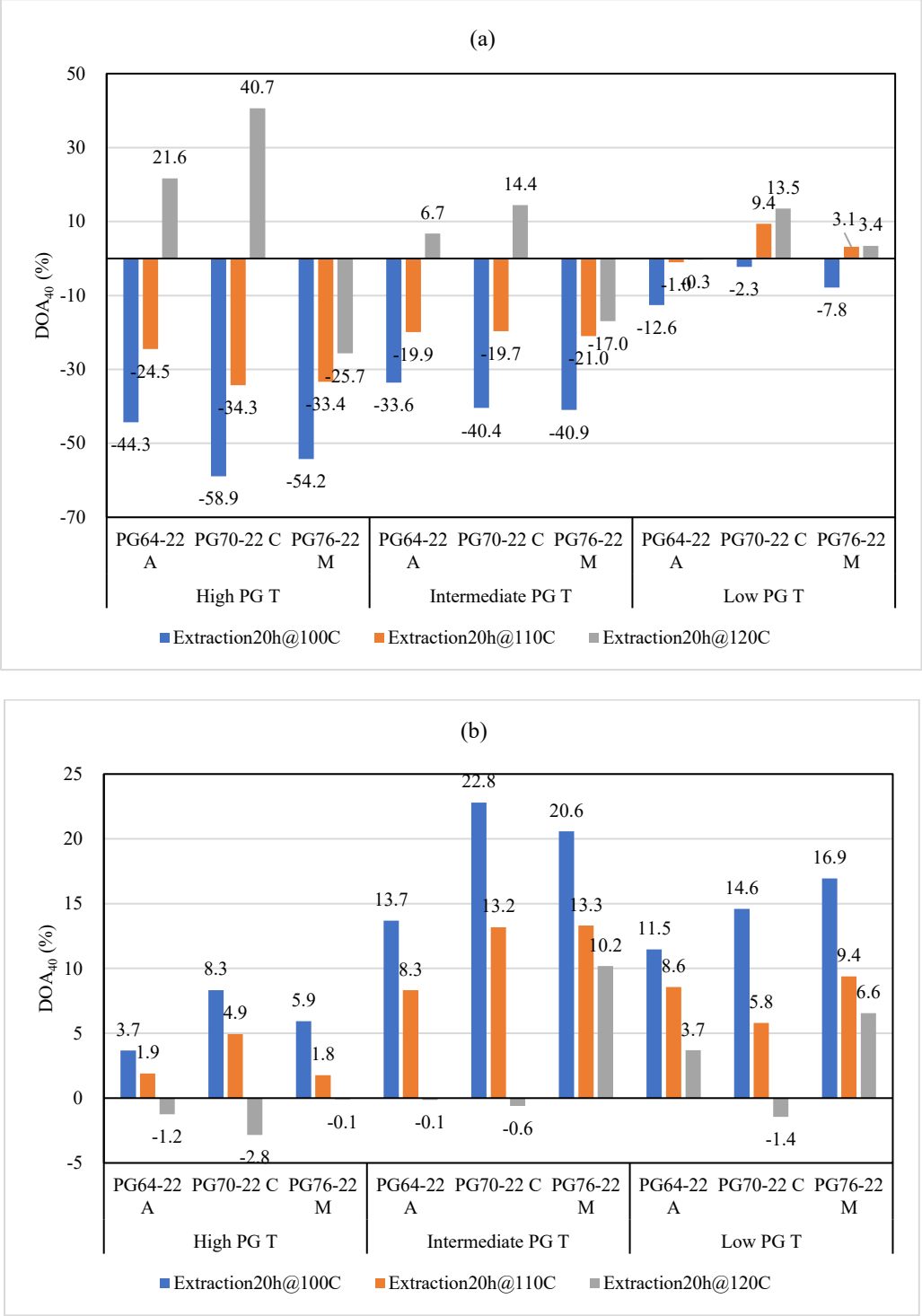


Figure 6.22 DOA_{40} for DSR parameters at three PG temperatures: (a) G, (b) δ.*

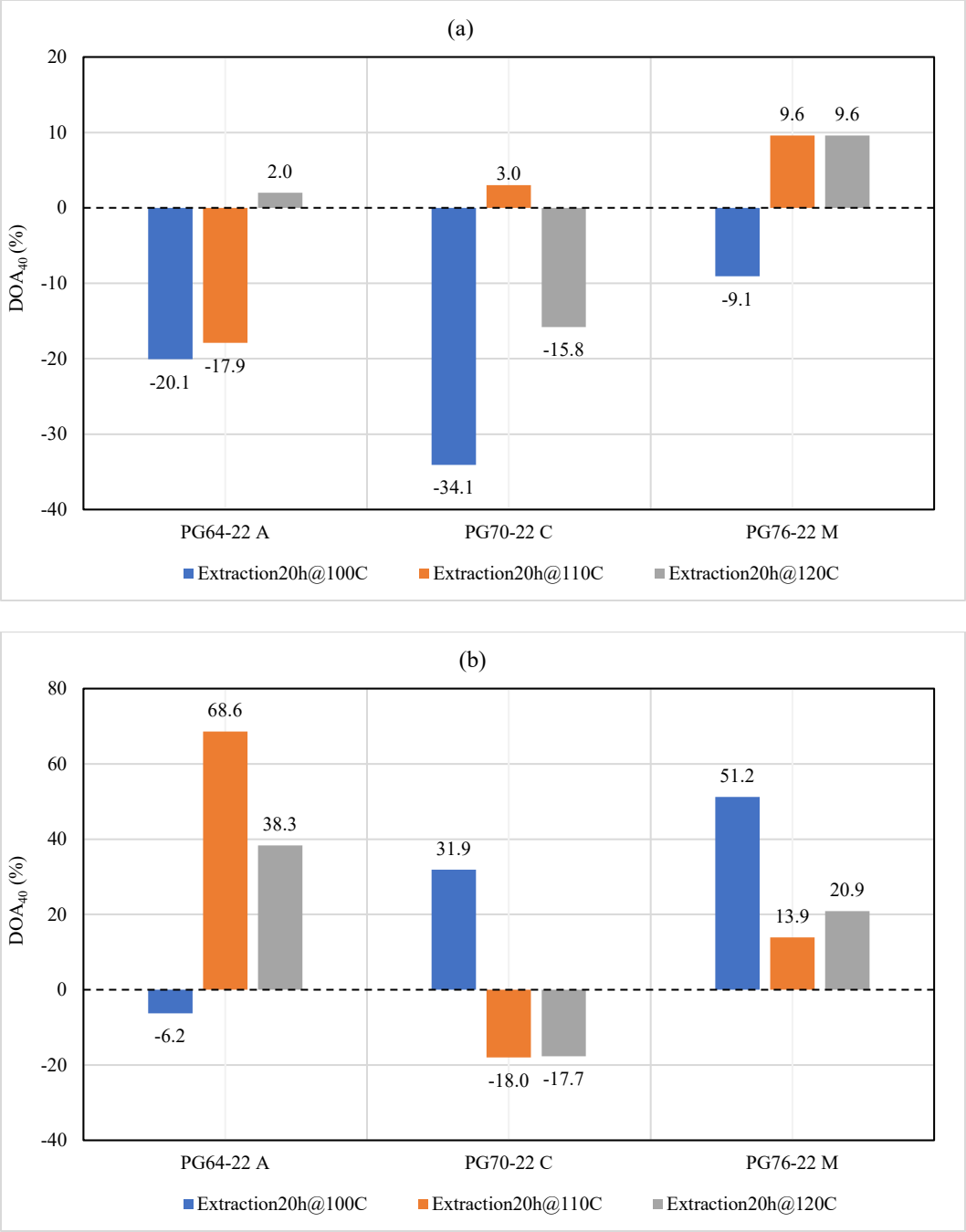


Figure 6.23 DOA_{40} for Poker-chip test parameters: (a) Peak stress, (b) Ductility.

6.2.3.2. Effect of the long-term aging on asphalt mixture cracking resistance

The testing results for mixtures with PG64-22, PG70-22, and PG76-22 binders are shown in Figure 6.24 and Figure 6.25. The data indicates the following trends:

1. The CTindex decreased, and CPR increased as the aging term progressed and aging temperature increased, showing that the mixture's cracking resistance weakened with aging.

2. The aging rate varies among these three mixtures, since the slopes of the mixtures versus aging temperature are different. The mixture aging rate is sensitive to aging temperature. For example, the mixture with PG64-22 and 20% RAP has a higher aging rate when the aging temperature increased from 100°C to 120°C.

3. Furthermore, the ranking of mixture cracking resistance at the short-term aging is different from that at the long-term aging, as shown by the lines crossing over each other in Figure 6.24 and Figure 6.25. Therefore, mixture long-term aging must be considered at mix design and even in the stage of mix plant production.

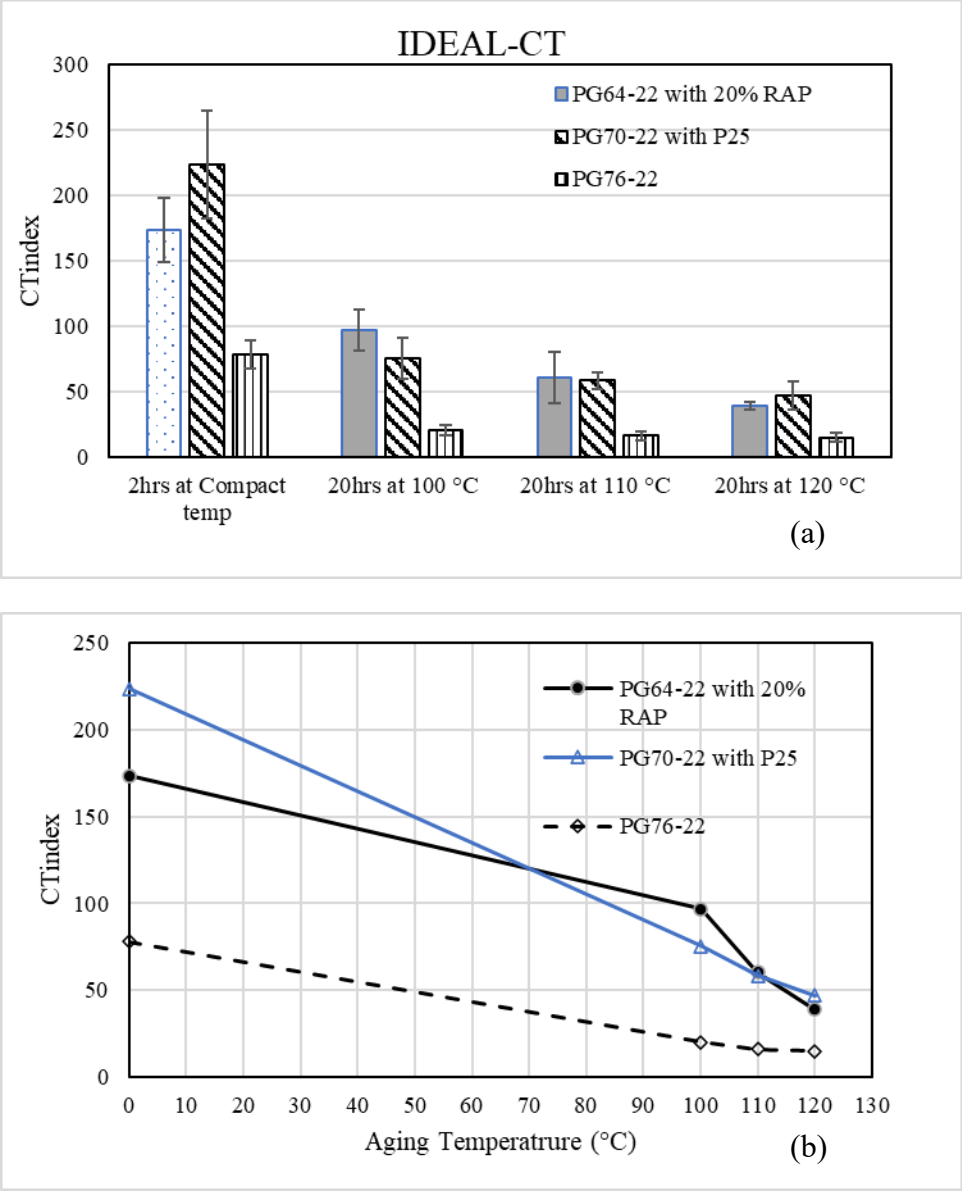


Figure 6.24 IDEAL-CT testing results at multiple aging conditions: (a) Bar chart, (b) Line chart.

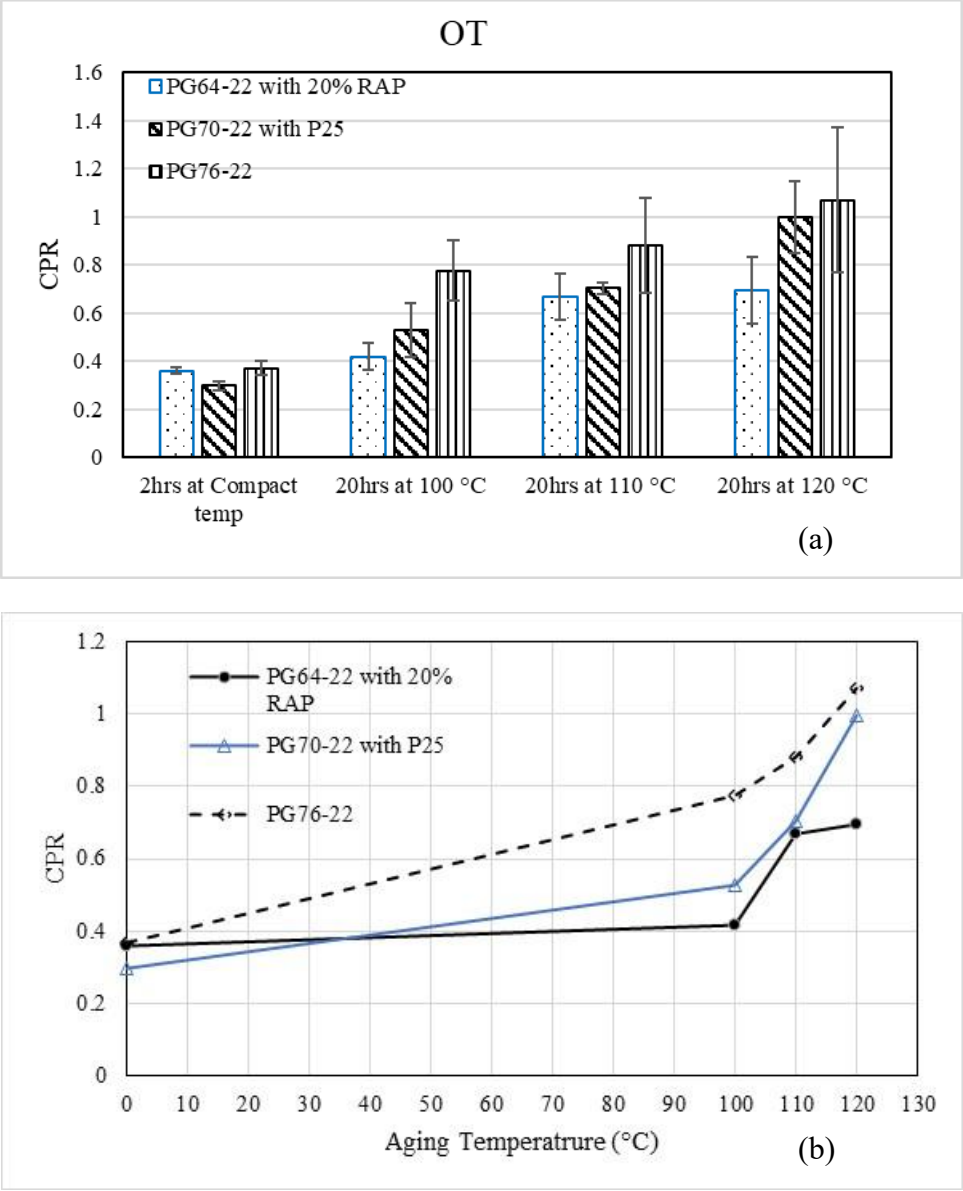


Figure 6.25 OT testing results at multiple aging conditions: (a) Bar chart, (b) Line chart.

6.2.4. Summary

In this section, the accelerated long-term aging protocol for asphalt mixtures was presented. Binder extraction and recovery tests were used to extract asphalt binders from loose mixtures, and DSR and poker chip tests were conducted to investigate their performance relationship with laboratory-aged binders. Subsequently, IDEAL-CT and OT cracking tests were employed to evaluate the impact of long-term aging on the cracking performance of asphalt mixtures.

The results indicate that increasing the aging temperature of loose mixes leads to a more severe aging level of the asphalt binder. Extraction 20hr. at 100°C can result in G* across different temperatures, high-temperature δ, and poker-chip ductility that are more comparable to those of

PAV20. Extraction 20hr. at 110°C produces intermediate- and low-temperature δ closer to PAV20. Extraction 20hr. at 120°C generally yields rheological and cracking parameters that align more closely with those of PAV40. The results also clearly demonstrate that long-term aging significantly degrades the cracking resistance of asphalt mixtures, with increased aging time and temperature correlating to lower CTindex values and higher CPR values. It also clearly shows the consideration of long-term aging asphalt mixes at the stages of mix design, trial batch, and plant production is necessary. To practically address the impact of long-term aging on asphalt mix properties, a representative long-term aging was recently recommended by researchers [100].

Chapter 7. Summary and Conclusions

This work began with the main goal of identifying and updating the tests and specifications in Item 300 as needed to ensure that the test methods and specifications are relevant, not redundant, and up-to-date.

Chapter 2 describes a review of the current test procedures for relevance, applicability, accuracy, and practicality, as well as recommendations for improving the tests. Recommended changes along with the adoption level were developed.

Chapter 3 developed new test procedures as identified to fulfill needed changes. Test procedures were developed in TxDOT format and included in appendices.

Chapter 4 generated and analyzed data collected for the new test procedures developed in this project. This data explored the agreement between laboratories, developed test conditions, and round-robin experiments. This data helped inform decisions for recommended specification limits for the new tests.

Chapter 5 suggests changes to 2024 Item 300 (with inclusion of existing Special Provision 300-003). A revised draft of each table in Item 300 is presented, with a discussion on the changes. The revised draft of Item 300 specifications has been organized based on the tables in the current Item 300 specifications.

Chapter 6 details an extra study of binder aging of PG binders. This includes short-term aging and PAV20, which are currently included in the specifications, an even longer-term aging termed PAV40, and a new procedure termed Thin Film PAV20. The Thin Film PAV20 was found to produce similar binder aging as the PAV40, but only requiring 20 hours of aging time. This Thin Film PAV20 is outlined in a proposed test procedure, based on the Pressure Air Vessel with modified test and specimen conditions.

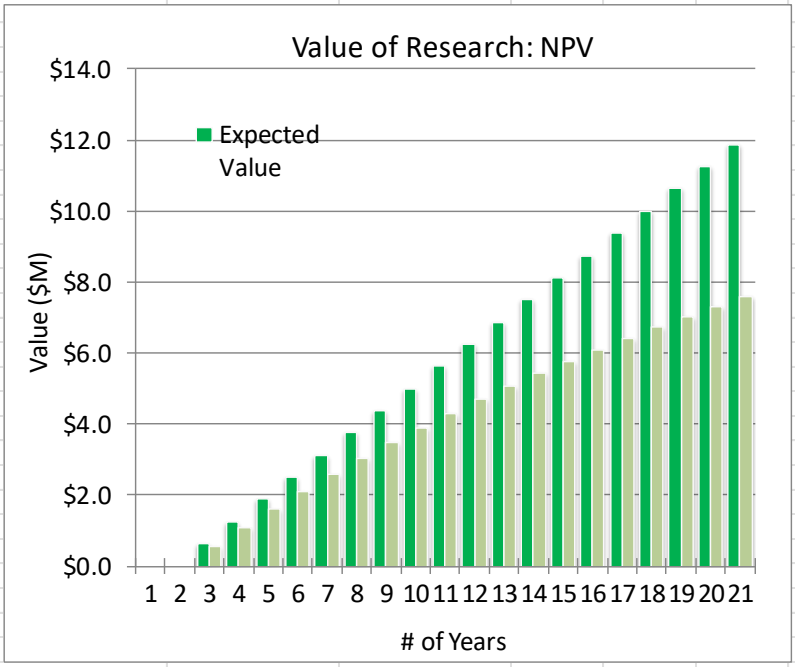
This work is presented to the Receiving Agency for their evaluation and possible update of Item 300, “Asphalts, Oils, and Emulsions.”

Value of Research

This Value of Research is based on savings generated by reduction in equipment diversity (equipment cost, maintenance, and calibration), reduction in waste generation, decreased testing time, and inclusion of more performance-related tests. This is estimated at $650,000 per year.

Texas Department of Transportation	**Project #**	0-7073		
	Project Name:	Improving Testing Requirements in Item 300 of TxDOT Standard Specification		
	Agency:	CTR & TTI	**Project Budget**	$ 1,130,000
	Project Duration (Yrs)	5.0	**Exp. Value (per Yr)**	$ 625,000
	Expected Value Duration (Yrs)	10	**Discount Rate**	4%
Economic Value				
	Total Savings:	$ 4,495,000	**Net Present Value (NPV):**	$ 4,296,489
	Payback Period (Yrs):	1.808000	**Cost Benefit Ratio (CBR, $1 : $___):**	$ 3.80

Years	Expected Value
0	$0
1	$0
2	$625,000
3	$625,000
4	$625,000
5	$625,000
6	$625,000
7	$625,000
8	$625,000
9	$625,000
10	$625,000
11	$625,000
12	$625,000
13	$625,000
14	$625,000
15	$625,000
16	$625,000
17	$625,000
18	$625,000
19	$625,000
20	$625,000
21	$0
22	$0
23	$0
24	$0
25	$0
26	$0
27	$0
28	$0
29	$0
30	$0



Notes:

Amounts on Value of Research are estimates.

Project cost should be expensed at a rate of no more than the expected value per year.

This electronic form contains formulas that may be corrupted when adding or deleting rows, by variables within the spreadsheet, or by conversion of the spreadsheet. The university is responsible for the accuracy of the Value of Research submitted.

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Appendix A

Test Procedure for

MEASUREMENT OF POLYMER SEPARATION ON HEATING IN MODIFIED ASPHALT SYSTEMS

TxDOT Designation: Tex-540-C

Effective Date: Draft

1. SCOPE

1.1. This procedure describes a method of predicting the degree of phase separation such as settlement that may occur when modified asphalt systems (those containing latex rubber, SBS block copolymer, tire rubber, or other modifiers) are heated and stored.

1.2. The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

2. APPARATUS

2.1. *All equipment necessary*, to perform the testing described in ASTM D7173.

2.2. *All equipment necessary*, to perform the testing described in AASHTO T 315.

3. PROCEDURE

3.1. Prepare the samples from the top and bottom portion of aluminum tubes as described in ASTM D7173.

3.2. Perform dynamic shear rheometer tests on the top and the bottom materials using 25-mm parallel plate geometries in accordance with AASHTO T 315 and determine their G*/sin δ values at 12% strain, 10 rad/sec by averaging the last 10 out of 20 cycles. Use the test temperature as follows:

- 58°C for PG 58-XX,
- 64°C for PG 64-XX and Polymer-Modified Asphalt Cements as identified in Item 300 Table 3,
- 70°C for PG 70-XX,
- 76°C for PG76-XX, and
- 82°C for PG 82-XX.

3.3. Calculate the difference in top and bottom results (*D*) as a percentage of the average values.

- $D = [(G^*/\sin\delta)_{top} - (G^*/\sin\delta)_{bottom}]*100 / [(G^*/\sin\delta)_{top} + (G^*/\sin\delta)_{bottom}]/2$

3.4. Report results (in the nearest of 0.1) as:

- fail if $D > 10.0\%$, or
- pass if $D \leq 10.0\%$.

3.5. Repeat Sections 3.1 through 3.4 using any other test procedure appropriate or that is called for by the specific material specification.

Appendix B

Test Procedure for

RECOVERY OF ASPHALT RESIDUE FROM ASPHALT EMULSION AND CUTBACK ASPHALT BY VACCUM OVEN

TxDOT Designation: Tex-55C-C

Effective Date: Draft

1. SCOPE

1.1. Use this procedure to recover asphalt residue from asphalt emulsions and cutback asphalts. Residue may be subsequently evaluated for material characteristics.

1.2. The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

2. APPARATUS

2.1. Vacuum oven. An example vacuum oven is shown in Figure 1.

2.1.1. Size commensurate with the number of samples for concurrent testing. Recovering emulsions and cutbacks in separate runs or separate ovens is a good practice.

2.1.2. Minimum vacuum rating of 80 mbar (60 mmHg) absolute pressure (vacuum).

2.1.3. Capable of maintaining 70°C +/- 2°C for emulsion recovery and 140°C +/- 2°C for cutback asphalt recovery.

Note 1. Examples of a vacuum oven can be found at:
https://hydrobuilder.com/across-international-accutemp-0-9-cf-vacuum-oven-5-sided-heat-sst-tubing-ul-csa-certified.html?opts=eyJhdHRyaWJ1dGU5MDciOiI5Mzg4In0=
and
https://www.acrossinternational.com/250c-ul-certified-1-9-cf-vacuum-oven-5-sided-heat-sst-tubing.html

Figure 1. An example vacuum oven

2.2. Vacuum Pump. Capable of providing at least 60 torr (80 mbar) of absolute pressure (vacuum).

Note 2. Examples of a vacuum pump can be found at:
https://www.fishersci.com/shop/products/welch-dryfast-tuneable-chemical-duty-vacuum-pumps-100-c-boiling-point-solvents-4/01257510?keyword=true
and
https://www.fishersci.com/shop/products/welch-dryfast-tuneable-chemical-duty-vacuum-pumps-100-c-boiling-point-solvents-4/01257502

2.3. Volatiles trap to prevent water (from emulsions) and solvent (from cutback asphalt) from entering the vacuum pump.

2.4. Sample containers for use with asphalt emulsions. Aluminum Weighing Dishes, 140 ml capacity, 3.75-inch (9.525 cm) base I.D, 0.75-inch (1.905 cm) height. Tests are performed in duplicate, so at least 2 samples containers are required per sample. An example is shown in Figure 2.

Note 3. Examples of a sample container can be found at: https://www.mcmaster.com/17805T82/

Figure 2. Aluminum sample dishes for asphalt emulsion.

2.5. Sample containers for use with cutback asphalt. Use round , 4oz, metal tins, approximately 2.375 inch (6 cm) inside diameter and 1-11/16 inch (4.3) cm height, as shown in Figure 3 (no lids are used). Tests are performed in duplicate, so at least 2 tins are required per sample.

Note 4 An example of 4 oz metal tin can be found at: https://www.uline.com/Product/Detail/S-17906SIL/Retail-Boxes/Deep-Metal-Tins-Round-4-oz-Solid-Lid-Silver

Figure 3. 4oz Metal Tins.

2.6. Syringes for transferring samples into the molds. A 10 ml syringe has been found adequate.

2.7. Spatula for stirring asphalt samples to ensure uniformity.

2.8. Balance, Class G1 in accordance with Tex-901-K, minimum capacity of 500 g, and the ability to hold a sample mold.

3. MATERIALS

3.1. Asphalt emulsion.

3.2. Cutback asphalt.

4. PROCEDURE

4.1. Set the vacuum oven to 70°C for asphalt emulsion and 140°C for cutback asphalt. Preheat the oven for a minimum of 12 hours to ensure temperature consistency and uniformity. Ensure the oven is at the proper temperature before proceeding.

4.2. Determine the vacuum gage setting needed.

4.2.1. If the vacuum oven pressure gage reads absolute pressure, the setting should be for 90 mbar +/- 10 mbar (67.5 mmHg +/- 7.5 mmHg) absolute pressure for testing.

4.2.2. If the vacuum oven pressure gage reads relative to atmospheric pressure, use the following equation to determine the pressure setting to use for testing.

$$P_{[v\text{-}gage]} = P_{[atm]} - P_{[set]}$$

Where $P_{[v\text{-}gage]}$ is the vacuum oven pressure gage setting,
$P_{[atm]}$ is the current ambient barometric pressure, and

$P_{(test)}$ is the absolute pressure desired for testing.

4.3. For asphalt emulsion:

4.3.1. Stir the emulsion thoroughly for 1 minute using a spatula.

4.3.2. Weigh the sample container and record the mass (A).

4.3.3. Tare the balance and transfer 5.20 g +/- 0.05 g of asphalt emulsion to the sample container. Record the mass poured in the mold (B).

4.3.4. Repeat 4.3.2 and 4.3.3 to generate a total of two duplicate samples for residue recovery.

4.3.5. Cap the emulsion container immediately after transferring the emulsion into the sample container to ensure minimum loss of water from container for future use.

4.3.6. Tip the sample container from side to side to spread the emulsion in the mold to get a uniform thickness of the emulsion layer.

4.3.7. While spreading the emulsion in the sample container, keep the walls of the mold clean.

4.3.8. Place the sample containers in the oven at 70°C +/- 2°C and apply vacuum for 2 hours.

4.3.9. Start the timer once the target vacuum pressure is reached and do not open the door during the test.

4.3.10. After 2 hours +/- 5 minutes under vacuum, release the vacuum in no more than 5 minutes.

4.3.11. Remove the samples from the oven and immediately weigh the sample containers and record the mass (C) to calculate the mass loss.

4.3.12. If residue testing is desired, scrape the residue from the sample container using a spatula and test the residue within 24 hours from when the samples are removed from the oven.

4.4. For cutback asphalt:

4.4.1. Stir the cutback asphalt thoroughly for 1 minute using a spatula.

4.4.2. Weigh the empty sample container and record the mass (A).

4.4.3. Tare the balance and transfer the required amount of cutback asphalt to sample container. Record the mass poured in the mold (B).

The sample amount is different for each viscosity grade of cutback to adjust for the binder content. Use the following sample amounts:

MC-30 use 2.70 g +/- 0.05 g.

RC-250 or MC-250 use 2.00 g +/- 0.05 g.

MC-3000 use 1.80 g +/- 0.05 g (tentatively included in this procedure).

MC-800 use 2.00 +/- 0.05 g (tentatively included in this procedure).

4.4.4. Repeat 4.4.2 and 4.4.3 to generate a total of two duplicate samples for residue recovery.

4.4.5. Cap the cutback asphalt container immediately after transferring the cutback asphalt into the sample container to ensure minimum loss of solvent from container for future use.

4.4.6. Tip the sample container from side to side to spread the cutback to get a uniform thickness of the cutback asphalt layer. Slight warming may be necessary for higher viscosity grades.

4.4.7. While spreading the cutback asphalt in the sample container, keep the walls of the pan clean.

4.4.8. Place the sample containers in the oven at 140°C +/- 2°C and apply vacuum for the specified time.

4.4.9. For Rapid Curing Cutbacks use 3 hours of vacuum oven curing time. For Medium Curing Cutbacks use 4 hours of vacuum oven curing time.

4.4.10. Start the timer once the target vacuum pressure is reached and do not open the door during the test.

4.4.11. After the specified curing time under vacuum, release the vacuum in no more than 5 minutes.

4.4.12. Remove the samples from the oven and immediately weigh the sample containers and record the mass (C) to calculate the mass loss.

4.4.13. If residue testing is desired, scrape the residue from the sample container using a spatula and test the residue within 24 hours from when the samples are removed from the oven.

5. REPORT

5.1. Report the mass loss (%) as ((C-B-A)*100/B), to the nearest 0.1 percent.

Appendix C

Test Procedure for

VISCOSITY DETERMINATION OF ASPHALT EMULSION AND CUTBACK ASPHALT USING ROTATIONAL VISCOMETER

TxDOT Designation: Draft

Effective Date: Draft

1. SCOPE

1.1. This procedure outlines the procedure for measuring the viscosity of asphalt emulsions and cutback asphalt using a Rotational Viscometer apparatus.

1.2. The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

2. APPARATUS

2.1. *All equipment necessary,* to perform the testing described in AASHTO T316.

3. PROCEDURE

3.1. Read and understand the information in the rotational viscometer manufacturer's operating manual before proceeding.

3.2. Prepare and calibrate the apparatus as described in ASTM D7173.

3.3. Preheat the sample holder with the sample chamber and the selected cylindrical spindle #18 according to the manufacturer's recommendation.

3.4. Set the proportional temperature controller to 60°C.

3.5. When the proportional temperature controller reads 60°C, remove the sample holder, and pour the required amount of asphalt emulsion/cutback asphalt as recommended by the manufacturer in the sample holder for testing.

3.6. Insert the sample holder into the temperature controller unit.

3.7. Insert a preheated spindle and attach it to the viscometer using the necessary coupling. Gently lower the spindle into the asphalt emulsion/cutback asphalt, so that the binder covers the upper conical portion of the spindle. This procedure may vary based on the manufacturer's recommendations.

3.8. Bring the asphalt emulsion/cutback asphalt sample to 60°C within approximately 30 min. Set the viscometer speed at 20 rpm and set the display to read the viscosity in centi-Poise (cP). The viscometer speed may be set higher than 20 rpm if it is expected that the observed torque will be out of range at 20 rpm.

3.9. Allow the sample to equilibrate at the desired test temperature for a minimum of 10 min. Begin the spindle rotation during the 10-min temperature equilibration period. Allow the readings to stabilize before recording any viscosity measurements. If the observed torque is out of range for the selected spindle and speed, change the spindle or speed based on the manufacturer's recommendations for the anticipated viscosity. If a different spindle is used, restart the test with a new sample.

3.10. Start the test after the asphalt binder sample has reached the specified temperature and equilibrated and the viscosity readings have stabilized, as required in the previous two steps.

3.11. Measure the viscosity at 1-min intervals for a total of 3 min.

---

4. REPORT

4.1. Report the following information:

- The date and time of the test;
- The test temperature to the nearest 1°C;
- The speed in rpm;
- The size of the spindle used;
- The torque in percent; and
- The average viscosity in cP.

Appendix D

Test Procedure for

LOW TEMPERATURE DSR TESTING OF ASPHALT BINDER

TxDOT Designation: Tex-55A-C

Effective Date: Draft

1. SCOPE

1.1. Use this test method, based on AASHTO T315, to evaluate the low temperature properties of binders with a Dynamic Shear Rheometer (DSR). This procedure extends T315 after DSR intermediate temperature testing using PAV aged binder to measure G* and δ at low PG grade temperature. Low temperature limits on G* and δ are used to determine specification compliance instead of S and m from the Bending Beam Rheometer (BBR).

1.2. The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

2. APPARATUS

2.1. Use all apparatus described in AASHTO T315 with a DSR that can condition and test samples at low PG temperature plus 10°C. Additionally, the DSR should have the ability to adjust the axial force to accommodate the temperature transition from intermediate to low temperature. For measurements at low temperature, DSR machine compliance may be not a concern if operating in a range of less than 20% of maximum torque. Typical DSRs with a maximum torque of 200 mN-m will be operating at approximately 10% of the maximum torque.

3. MATERIALS

3.1. Test asphalt binders that have been conditioned with the RTFO and PAV-20hr to simulate a long-term aging state.

4. PROCEDURE

4.1. Preheat the DSR temperature chamber to 70 °C.

4.2. Transfer the asphalt binder sample to one of the test plates and test the sample at intermediate temperature according to AASHTO T315.

After completion of 8 mm intermediate temperature testing and without removing the sample, lower the temperature from the intermediate temperature to the low PG temperature plus 10°C and use the axial force adjustment at 0.3N (+/- 0.1N) in compression.

Note: The axial force adjustment is required to accommodate the volume change of the specimen as the temperature is reduced from intermediate to low temperature to prevent debonding of the specimen from the DSR plate or platen.

4.3. Condition the sample at the test temperature (low PG grade temperature plus 10°C) for 2100 s.

4.4. Test the specimen at 0.1% strain rate and 0.2 rad/s angular frequency for 10 cycles.

5. REPORT

5.1. Report the average G* and δ from the last 5 cycles at the PG low temperature grade plus 10°C.

Appendix E

Test Procedure for

POKER CHIP TEST OF ASPHALT BINDER

TxDOT Designation: TEX-55B-C

Effective Date: Draft

1. SCOPE

1.1. This test method evaluates the tensile failure characteristics of an asphalt binder under a state of stress that induces high triaxiality in the asphalt binder, which models a realistic stress state similar to asphalt binder confined between two aggregates. This test method is relevant for fatigue or thermal cracking in asphalt mixtures and can be used with material aged using T 240 (RTFOT) and R 28 (PAV).

1.2. The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

2. APPARATUS

2.1. *Testing Machine* – The testing machine or load frame, as shown in Fig. 1, should have the capability of applying tension in displacement-controlled mode, then switching to load-controlled mode until the sample fails, and recording load and displacement during this time.

Note 1 – A servo hydraulic testing machine with a function generator capable of producing the desired displacement-controlled or load-controlled mode has been shown to be suitable for use in tension testing. Other commercially available electric motor actuator and load frame with at least 900 lbf (4000 N) load cell can also be used.

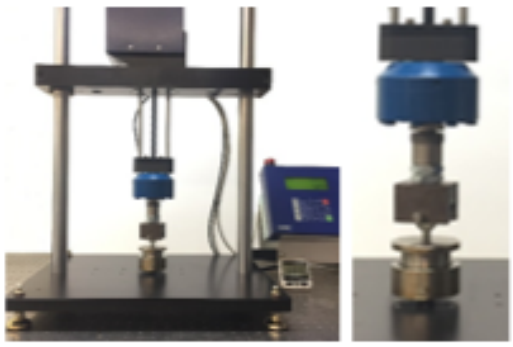

Figure 1 - Testing Machine (left: overall view of the load frame, right: closer view of the load cell).

Note 2 – The device should be capable of applying a displacement rate of at least 5.9 in/minute (150 mm/minute).

2.1.1. The load frame must be set up to apply tension in displacement-controlled mode of 0.04 in/minute (1 mm/minute) until a tensile load of 9 lbf (40 N) is reached. At this point the load frame must switch to a load-controlled mode to apply a tensile load of 0.25 lbf/second (2 N/second) until the sample fails. During this time, the load and displacement are recorded at a rate of at least 2 points per second.

2.1.2. The testing machine must be coupled with an automatic cut off system that can be programmed using a digital controller to stop the equipment in case of emergency.

2.2. *Measurement and Recording System* – The system should include sensors for measuring and recording vertical deformations. The system should be capable of measuring vertical deformations with a resolution of at least 0.00004 in. (0.001 mm). Loads should be measured and recorded with a resolution of at least 0.0225 lbf (0.1 N).

2.2.1. The vertical deformation can be measured by a linear variable differential transducer (LVDT) or other suitable devices. The sensitivity and type of measurement device should be selected to provide the deformation readout required in 2.2.

2.2.2. The load should be measured with an electronic load cell capable of satisfying the specified requirements for load measurements in 2.2.

2.2.3. Refer to the manufacturer's specifications for system calibration and accuracy.

2.3. *Temperature-Controlled Chamber for Conditioning Specimen* – The temperature-controlled chamber should be capable of maintaining a temperature of 77 ± 1°F (25 ± 0.5°C). The chamber should be large enough to hold at least ten specimens for a period of at most 24 h prior to testing.

2.4. *Oven* – The oven must be capable of maintaining a temperature of 320 ± 5°F (160 ± 3°C).

2.5. *Stainless-Steel Mold* – The poker chip sample is prepared using a two-part mold (top and bottom) and shall be similar in design to that shown in Fig. 2 and 3. The mold shall be made of stainless-steel, and the dimensions of the assembled mold shall be as shown in Fig. 2 and 3, with the permissible variations indicated. Two parts of the mold, as shown in Fig. 4, are used to prepare a thin film specimen.

Note 3 – Mold thread size and depth may vary based on the mounting system of the testing machine.

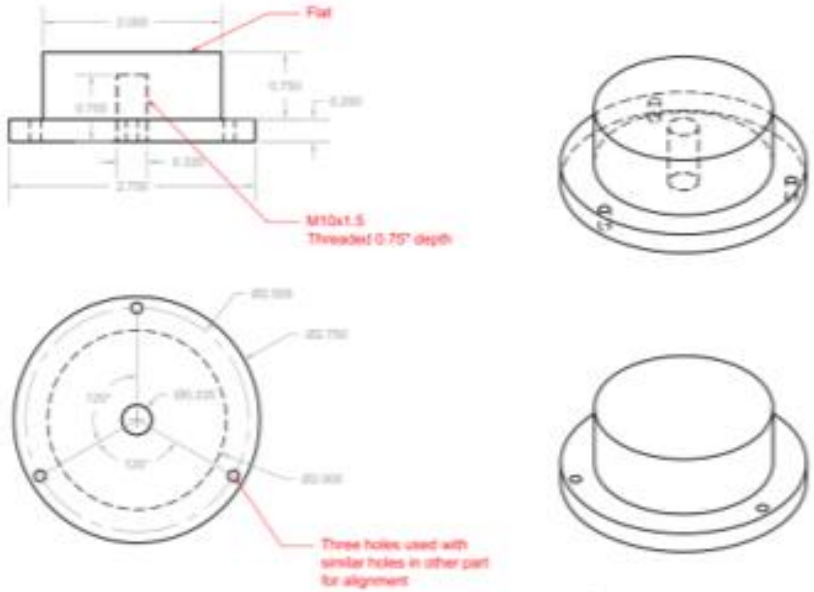

Figure 2 – Stainless-Steel Mold. Top part of the poker-chip mold (all dimensions in inches).

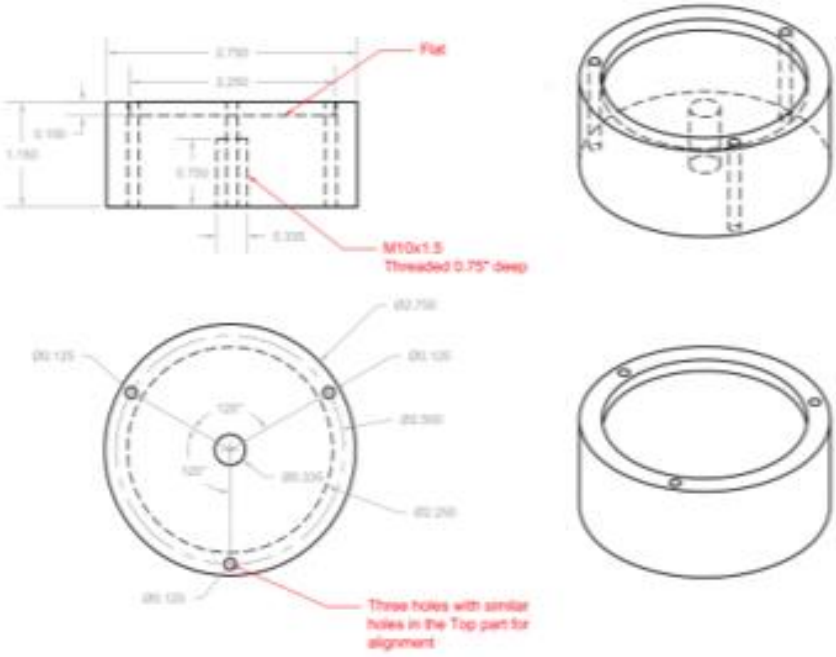

Figure 3 – Stainless-Steel Mold. Bottom part of the poker-chip mold (all dimensions in inches).

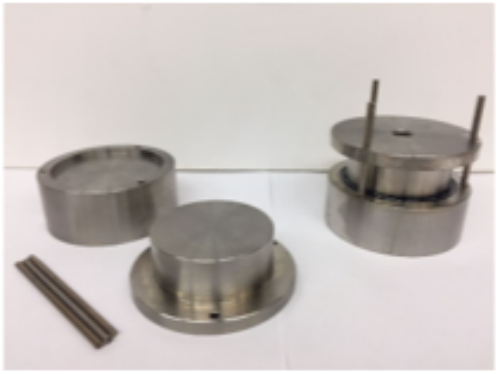

Figure 4 – Poker chip stainless-steel mold and laboratory-molded specimen between the top and bottom parts that are aligned using three alignment pins (top and bottom of the mold with the three alignment pins are shown to the left and an assembled mold with the binder sample is shown to the right).

2.6. *Alignment pins* – The alignment pins can be machined or purchased directly from a hardware supplier. The pins shall be made of stainless-steel and should be 0.122 ± 0.0002 in (3.1 ± 0.005 mm) diameter and 2 ¾ in (69.85 mm) length. These pins are run through the three holes in the top and bottom of the poker chip specimen mold to ensure proper alignment of the two parts. Three alignment pins are needed for each poker-chip mold.

2.7. *Spacer Dowels* – The spacer dowels can be machined or purchased directly from a hardware supplier. The spacer dowels shall be made of alloy steel and should be 1/16 ± 0.0003 in (0.0025 ± 1.18E-05 mm) diameter and 1/8 in (0.005 mm) length. These spacer dowels are dropped in the liquid asphalt binder to achieve the target thickness when placing the top part of the poker chip mold.

2.8. *Ball Joint Rod End* – The ball joint rod end can be machined or purchased directly from a hardware supplier and shall be made of alloy steel with a ball joint end type. This ball joint rod end is used on the top part of the poker chip mold to connect to the load frame using an appropriate clevis pin and dowel.

2.9. *Silicone Molds* – The silicone molds should be made of food grade silicone, bisphenol A (BPA) free, non-stick and flexible to easily release the asphalt binder from the molds. Typically, the temperature tolerance range of this material is from -40 to 466°F (-40 to 241°C). The dimensions of each cavity are 1.0 in (25.4 mm) diameter and 1/4 in (6.35 mm) depth. These dimensions hold approximately 0.159 oz (4.5 g) of asphalt binder properly.

2.10. *90 Degree Bent Nose Pliers* – The 90-degree bent nose plier is a variation of needle nose pliers. It features non-serrated tapered jaws that are bent at a 90-degree angle, 3/8" from the tip. This allows the jaws to grip the spacer dowels.

2.11. *Stainless-Steel Trays* – The stainless-steel trays should have a size of 13 x 9.5 x 1 in (330.2 x 241.3 x 25.4 mm) with a flat rigid base to accommodate up to 10 molds and carry them in and out of the oven or temperature-controlled chamber.

2.12. *Acetone* – Acetone can be used to clean and remove dust from the surface of the stainless-steel trays and silicone molds.

2.13. *Dry wipes* – Dry paper tissue wipes provide an easy way to wipe up liquid and dust from the surface of the stainless-steel trays and silicone molds.

2.14. *Balance* – Balance conforming to the requirements of AASHTO M 231, Class G2.

2.15. *Stainless-Steel Micro Spatula* – The stainless-steel spatula that is 22.5 cm (9.0 in) length and 5 x 0.78 cm (2 x 0.31 in) flat ends and mirror-like finish.

2.16. Bubble Level – The bubble level can be used to indicate whether the surface of the stainless-steel trays containing the poker-chip molds are horizontal (level).

3. MATERIALS

3.1. Asphalt Binder Specimen – The asphalt binder can be aged using T 240 (RTFOT) or R 28 (PAV), or both.

3.2. Recovered Specimen – The recovered asphalt binder from an asphalt mixture or reclaimed asphalt pavement (RAP) can be tested in an "as is" condition or it can be aged using R 28 (PAV).

4. SPECIMENS

4.1. *Laboratory-Molded Specimen* – Condition the asphalt binder in accordance with AASHTO T 240 or TxDOT Tex-541-C (RTFOT). If long term aged asphalt binder is to be tested, obtain test sample according to AASHTO R 28 (PAV). Two circular specimens of 2.0 in (50.8 mm) diameter with a 1/16 in (1.5875 mm) thickness, are prepared using the stainless-steel mold described in the section above.

Note 4 – The asphalt binder is heated inside an oven until the material is sufficiently fluid. Typically, a temperature of 320 ± 5°F (160 ± 3°C) is adequate. Modified asphalt binders may be heated to higher temperatures if material does not pour easily.

5. PROCEDURE

5.1. Asphalt Binder Sample Preparation:

5.1.1. Heat a 2 oz (60 ml) metal can, containing the asphalt binder sample, in an oven to 320 ± 5°F (160 ± 3°C) for 15 to 20 minutes until the binder is fluid enough to be poured. During the heating process, the sample should be covered.

5.1.2. Stir the sample thoroughly to ensure it is homogenous. Avoid introducing bubbles in the asphalt binder while stirring.

5.1.3. Pour 0.159 ± 0.0018 oz (4.5 ± 0.05 g) of the asphalt binder inside the silicon mold, as shown in Fig. 5, label the mold and allow it to cool at room temperature after pouring for a period of not less than 20 min. Prepare the poker chip sample within 48 hours of pouring.

Note 6 – The same amount of asphalt binder sample can be poured directly into a clean pre-heated bottom part of the poker chip mold. However, the balance used must be insulated to protect the weighing plate from the pre-heated bottom part of the poker chip mold.

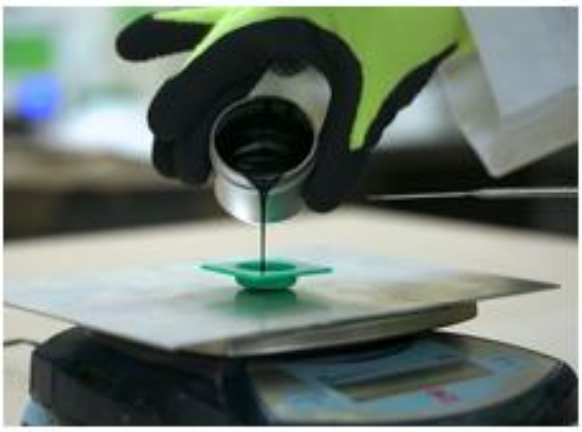

Figure 5 - Asphalt binder poured inside the silicone mold.

5.2. Laboratory-Molded Specimens Preparation:

5.2.1. Wipe and clean the top and bottom test faces of the poker chip mold using acetone.

5.2.2. Place the three alignment pins in the bottom part of the poker chip mold.

5.2.3. Preheat the top and bottom parts of the poker chip mold and the spacer dowels in a conventional oven at 320 ± 5°F (170 ± 3°C) for at least 3 hours (preheating overnight is recommended). The poker chip molds can also be left overnight in the oven.

Note 7 – Stainless-steel trays can be used to transport the poker chip molds. Ensure that the oven trays are level using a bubble level prior to use.

5.2.4. Remove the pre-weighed dollop of the asphalt binder from the silicon mold and place it in the bottom part of the poker chip mold. Place the bottom part of the mold back in the oven at 320 ± 5°F (170 ± 3°C) for 15 minutes or until the dollop of asphalt binder is completely molten and spread over the entire bottom surface of the poker chip mold.

5.2.5. Place three spacer dowels approximately at 120 degrees apart and about halfway radially out from the center of the bottom poker-chip sample disc, as shown in Fig. 6.

Note 8 – 90-degree bent nose pliers can be used to grip the spacer dowels.

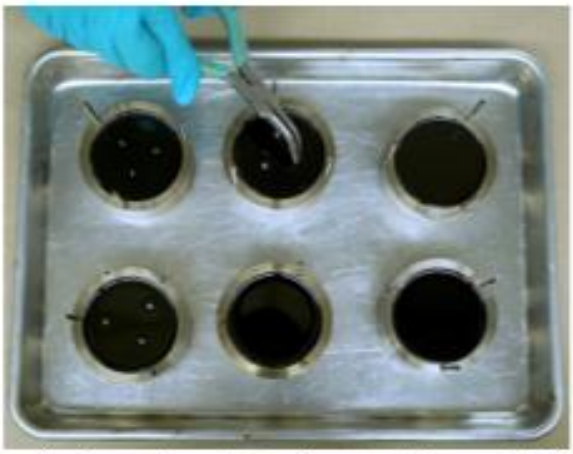

Figure 6 – Spacer dowels placed on top of the asphalt binder.

5.2.6. Place the bottom part of the poker chip mold back in the oven for another 15 to 20 minutes to allow the spacer dowels to sink and submerge in the binder.

Note 9 – The spacer dowels will be partially submerged due to the surface tension of asphalt binder.

5.2.7. Remove the bottom part and the pre-heated top part of the mold from the oven. Place two alignment pins in the lower part of the mold. Lower the top part of the poker chip mold slowly through the alignment pins and firmly press it at the center to ensure a correct set. Place the third alignment pin to ensure proper alignment.

Note 10 – Exercise caution. Plates are hot and the asphalt binder is fluid. Suddenly dropping the top poker-chip sample disc may cause the liquid binder to splash out.

5.2.8. Place the poker chip assembly in the oven at 320 ± 5°F (170 ± 3°C) for another 20 to 25 minutes.

5.2.9. Remove the poker chip assembly from the oven and let it cool to room temperature on a level platform for a period of not less than 30 min or until asphalt binder is completely solidified.

Note 11 – If the asphalt binder spilled over, discontinue the procedure and discard the specimen.

5.2.10. Place the poker chip assembly inside the temperature-controlled chamber at 77 ± 1°F (25 ± 0.5°C) until the specimen has reached the target temperature (conditioning overnight is recommended). Test the poker chip sample within 48 hours of conditioning. Ensure that the poker chip assembly is level using a bubble level.

Note 12 – If room temperature is within ± 1.0°F (± 0.5°C) of target temperature, the poker chip assembly can be cooled at room temperature.

5.3. Mounting and Testing Laboratory-Molded Specimens in the Testing Machine:

5.3.1. Ensure that the load cell of the testing machine is zeroed out with just the top part of the poker chip mold suspended from the loading axis.

5.3.2. Program the load frame to run the following sequence: i) apply tension in displacement-controlled mode of 0.04 in/minute (1 mm/minute) until a tensile load of 9 lbf (40 N) is reached, at this point ii) switch to load-controlled mode and apply a tensile load of 0.25 lbf/second (2 N/second) until the sample fails.

5.3.3. Take the specimen and gently remove the alignment pins while holding the bottom of the specimen.

Note 13 – Do not use force to remove the alignment pins; if they appear stuck for any reason, gently tap using the back of a screwdriver and try to wriggle it free.

5.3.4. Check the lip of the bottom of the specimen to ensure that there is no binder that spilled over; this would indicate that the specimen was not cooled on a level platform.

5.3.5. Lock the bottom of the poker chip assembly in the loading frame.

5.3.6. Thread in the ball joint rod on the top of the poker chip mold and ensure it is finger tight but do not apply too much torque to avoid damaging the specimen.

5.3.7. Lower the loading axis gently until the clevis rod end hole aligns with the hole in the ball joint rod. Gently slide a dowel through the clevis rod end and the ball joint rod locking the two in place. Once the sample is locked in place, run the programmed test.

5.3.8. Start the test using the programmed sequence according to section 5.3.2. The testing device automatically starts the test when the load frame has achieved a tensile load of 9 lbf (40 N).

5.3.9. Record the load and displacement at a rate of at least 2 points per second during the test. Fig. 7 shows a typical poker-chip test output data.

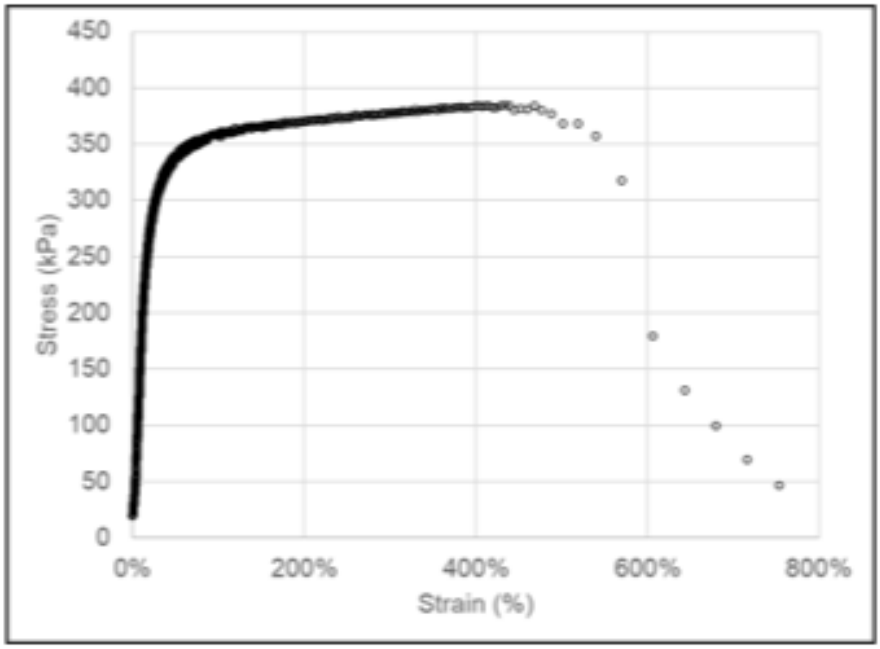

Figure 7 – Typical Poker-chip Test Output Data.

6. CALCULATIONS

6.1. Calculate the maximum stress, S_{Tmax}, recorded as the tensile strength as follows:

$$S_{Tmax} = \frac{4\,P_{ult}}{\pi D^2}$$

where:

P_{ult} = ultimate applied load required to fail specimen, lbf (or N), and

D = diameter of specimen, in (or mm).

6.2. Calculate the strain at 80% of the maximum stress, $\varepsilon_{0.8S_{Tmax}}$, as the ductility.

Note 14 – This parameter is calculated when 80% of the maximum tensile stress is reached after the ultimate applied load. Interpolate as necessary between data points.

$$\varepsilon_{0.8S_{Tmax}} = \frac{\delta}{t}$$

where:

δ = displacement at 80% after the ultimate applied load, in (or mm), and

t = thickness of specimen in (or mm).

7. REPORT

7.1. Report the following for each laboratory-molded specimen:

- asphalt binder grade;
- test temperature;
- maximum stress, S_{Tmax};
- ductility, $\varepsilon_{0.8S_{Tmax}}$;
- additional comments.

8. PRECISION

8.1. The precision of this test method has not been established.

9. FOR INFORMATION IN DRAFT DOCUMENT ONLY

9.1. The 3D parts for the mold, accessories and an instructional video can be found in the link below:

https://doi.org/10.18738/T8/KTCNVK

9.2. If you would like to order molds, these can be ordered from https://www.xometry.com

Please contact a-bhasin@mail.utexas.edu for more details on ordering from Xometry.