ASPHALT INSTITUTE®

July 2024

To: Roofing Asphalt Testing Laboratories

From: Wes Cooper, Laboratory Manager – Asphalt Institute

Re: Confirmation of Participation in the **2024** Proficiency Sample Program for Roofing Asphalts

Thanks for your interest in participating in the 2024 Proficiency Sample Program for Roofing Asphalts. This letter serves as a confirmation of your participation. If you requested us to bill your company, then an invoice will be sent by separate mailing.

Enclosed with this letter are the test samples and a sample data reporting sheet. There should be sufficient material in each container to conduct all of the listed tests including:

Softening Point, Ring & Ball	ASTM D36, *Test Method for Softening Point of Bitumen (Ring & Ball Apparatus)*
Softening Point, Cup & Ball	ASTM D3461, *Test Method for Softening Point of Asphalt and Pitch (Mettler Cup and Ball Method)*
Flash Point	ASTM D92, *Test Method for Flash and Fire Points by Cleveland Open Cup*
Penetration @ 0, 25, and 46°C (32, 77, and 115°F)	ASTM D5, *Test Method for Penetration of Bituminous Materials*
Ductility @ 25°C (77°F)	ASTM D113, *Test Method for Ductility of Bituminous Materials*
Rotational Viscosity @ 204°C (400°F)	ASTM D4402, *Test Method for Viscosity Determination of Asphalt at Elevated Temperatures Using a Rotational Viscometer*
Stain Index	ASTM D2746, *Test Method for Staining Tendency of Asphalt*
Rheological Testing – DSR	Draft Standard, *Test Method for Determining Rheological Properties of Roofing Asphalt Binders Using a Dynamic Shear Rheometer (DSR)*

We understand that not all the tests may be conducted on each sample by the participating lab. However, we would ask that each lab conduct as many tests as possible to allow for a complete analysis of the data.

Please note that we have added a question to the data reporting sheet asking if you stir the fluid when performing the softening point test. The ASTM method does not specify stirring, but requires a uniform heating rate. To accomplish this requirement, some labs have found it necessary to stir the fluid during the test.

The closing date to complete testing and submit test results to the Asphalt Institute for analysis is **October 11, 2024**. After the closing date, all data will be analyzed and a summary report will be submitted to each participant showing their individual lab results and a comparison with the rest of the labs. Since the proficiency program does not involve accreditation, it will be the participating lab's decision how to address test results that are more than two standard deviations from the mean. Scatter diagrams and QC charts are particularly helpful in visually identifying trends within a lab.

Please feel free to contact me (859-288-4983 or wcooper@asphaltinstitute.org) if you have any questions.

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ASPHALT INSTITUTE
2024 Roofing Asphalt Binder Proficiency Sample Program

Mail or e-mail this form to:
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wcooper@asphaltinstitute.org

Laboratory Name and Address:

Sample Preparation: Preheat the sample in an oven until fluid enough to pour. If necessary, remove any surface skin before pouring test samples.

Tests include: ASTM D92, *Test Method for Flash and Fire Points by Cleveland Open Cup*; ASTM D36, *Test Method for Softening Point of Bitumen (Ring & Ball Apparatus)*; ASTM D5, *Test Method for Penetration of Bituminous Materials;* ASTM D113*, Test Method for Ductility of Bituminous Materials;* ASTM D4402, *Test Method for Viscosity Determination of Asphalt at Elevated Temperatures Using a Rotational Viscometer*; ASTM D2746, *Test Method for Staining Tendency of Asphalt*; and ASTM D3461, *Test Method for Softening Point of Asphalt and Pitch (Mettler Cup-and-Ball Method)*.

Asphalt Binder Type: Roofing (Coating)	Sample 37	Sample 38
Standard Testing ***(see referenced ASTM standard test methods)***		
Flash Point, Cleveland Open Cup, °F ☐ Manual ☐ Automatic		
Softening Point, Ring and Ball, °F ☐ Manual ☐ Automatic Stirred during test? ☐ Yes ☐ No		
Softening Point, Mettler Cup and Ball, °F		
Penetration, 0°C (32°F), 200g, 60 sec, dmm *Room temperature during conditioning, °C:*		
Penetration, 25°C (77°F), 100g, 5 sec, dmm *Room temperature during conditioning, °C:*		
Penetration, 46°C (115°F), 50g, 5 sec, dmm *Room temperature during conditioning, °C:*		
Ductility, 25°C (77°F), cm		
Rotational Viscosity, 204°C (400°F), cP		
Spindle		
RPM		
Torque, %		
Stain Index		

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Asphalt Binder Type: Roofing (Coating)	Sample 37	Sample 38
***Optional Rheological Testing** (see instructions)*		
DSR, T_c where η* @ 1/rad/s = 1200 Pa-s, °C		
DSR, G* at 2.5 rad/s, 25°C, Pa		
DSR, δ at G*=10 MPa (10 rad/s), degrees		
DSR, Temperature at δ=27° (10 rad/s), °C		
DSR, G* at δ=27° (10 rad/s), °C		

Closing Date: October 11, 2024	
Note: Each individual test should be performed on both samples by the same operator, but it is not necessary that **all** tests be performed by the same operator	
Date(s) Sample Tested:	Reported by:
Tested by:	Lab Telephone:
e-mail:	FOR AI INTERNAL USE

Comments:

2024

To: Roofing Asphalt Testing Laboratories

From: Wes Cooper, Laboratory Manager – Asphalt Institute

Re: Optional Rheological Testing for the 2024 Proficiency Sample Program for Roofing Asphalts

Thanks for your interest in participating in the 2024 Proficiency Sample Program for Roofing Asphalts. For the past several years, we have offered labs the opportunity to conduct and report results from two optional rheological tests. Past research has indicated that these rheological tests could be correlated with softening point and penetration. We are continuing to include these two optional tests as part of the program.

A third optional rheological test has been included for 2024. This additional procedure is a temperature sweep intended to measure the stiffness and relaxation properties of roofing coatings at intermediate-low temperatures, and is currently being used by the Asphalt Institute Roofing Technical Advisory Committee Rheological Task Force in expoloratory work. Inclusion of this procedure in the 2024 program allows laboratories to gain experience with the testing and terminology, as well as assist in measuring the reproducibilty of the associated data outputs.

We understand that not all labs have the Dynamic Shear Rheometer (DSR) equipment and/or will want to conduct these additional tests. If your lab is interested in conducting these optional tests, please follow the general procedures described below. Also attached is a draft standard for your reference.

High Temperature DSR – Determination of T_c where η* = 1200 Pa-s

- Generally follow the equipment and specimen preparation procedures as described in ASTM D7175, *Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer*.
- Use 25-mm parallel plate geometry with a 1-mm gap.
- Load, trim the sample at 90°C. Allow the sample to achieve temperature equilibrium at 90°C before testing.
- Start testing at 90°C in oscillatory mode using 5% shear strain and a loading frequency of 1 rad/s. Determine the complex viscosity, η*, in Pa-s.
- Without removing the sample, increase the temperature to 100°C. Allow the sample to achieve temperature equilibrium at 100°C before testing.
- Start testing at 100°C in oscillatory mode using 5% shear strain and a loading frequency of 1 rad/s. Determine the complex viscosity, η*, in Pa-s.
- Without removing the sample, increase the temperature to 110°C. Allow the sample to achieve temperature equilibrium at 110°C before testing.
- Start testing at 110°C in oscillatory mode using 5% shear strain and a loading frequency of 1 rad/s. Determine the complex viscosity, η*, in Pa-s.
- Unload the sample and clean the DSR.
- Plot complex viscosity, η*, as a function of temperature. Determine the temperature, to the nearest 0.1°C, where η* is equal to 1200 Pa-s. This temperature may be interpolated or extrapolated.

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Intermediate Temperature DSR – Determination of $G^*_{2.5}$ at 25°C

- Generally follow the equipment and specimen preparation procedures as described in ASTM D7175, *Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer*.
- Use 8-mm parallel plate geometry with a 2-mm gap.
- Set the gap at 25C. Increase the temperature to 75°C and load, trim the sample. Lower the geometry to the testing gap (2 mm). Reduce the temperature to 25°C and allow the sample to achieve temperature equilibrium before testing.
- Start testing at 25°C in oscillatory mode using 0.7% shear strain and a loading frequency of 2.5 rad/s. Determine the complex modulus, G*, in Pa.
- Unload the sample and clean the DSR.

Intermediate-Low Temperature DSR – Determination of $\delta_{G^*=10\ MPa}$, log $G^*_{\delta=27°}$, and $T_{\delta=27°}$

- Generally follow the equipment and specimen preparation procedures as described in ASTM D7175, *Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer*.
- Use 8-mm parallel plate geometry with a 2-mm gap.
- Set the gap at 25C. Increase the temperature to 75°C and load, trim the sample. Lower the geometry to the testing gap (2 mm).
- Perform a temperature sweep in oscillatory mode using 0.1% shear strain and a loading frequency of 10 rad/s.
    - Perform the temperature sweep on the same sample in decreasing temperature steps at intervals of no greater than 6°C, allowing 10 minutes for thermal equilibrium at each test temperature. Test temperatures should be chosen such that complex modulus (G*) of 10 MPa (log G* of 7) and phasre angle (δ) of 27° will be bracketed.
    - Perform the test at each test temperature for sixteen loading cycles. Determine G* in Pa and δ in degrees as the average of the last eight loading cycles.
- Plot the logarithm of complex modulus, G*, and temperarure, T, as a function of phase angle, δ. Interpolate to determine:
    - The phase angle where log G* is 7 (i.e. G*=10 MPa), $\delta_{G^*=10\ MPa}$.
    - The logarithm of the complex modulus where phase angle is 27 degrees, log $G^*_{\delta=27°}$.
    - The temperature where phase angle is 27°, $T_{\delta=27°}$.
- Unload the sample and clean the DSR.

Please feel free to contact me (859-288-4983 or wcooper@asphaltinstitute.org) if you have any questions.

Standard Test Method for

Determining Rheological Properties of Roofing Asphalt Binders Using a Dynamic Shear Rheometer (DSR)

1. Scope
   1.1. This test method covers the determination of the dynamic shear modulus (G*) and phase angle (δ) of roofing asphalt binders when tested in dynamic (oscillatory) shear using parallel plate geometry.
   1.2. The values stated in SI units are regarded as standard.
   1.3. This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents
   2.1. ASTM Standards

   D5 Test Method for Penetration of Bituminous Materials

   D36 Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus)

   D7175 Test Method for Determining the Rheological Properties of Asphalt Binder using a Dynamic Shear Rheometer

3. Terminology
   3.1. Refer to ASTM D7175, Section 3 for relevant terminology.

4. Summary of the Test Method
   4.1. This standard includes the procedure to measure complex shear modulus (G*) and phase angle (δ) of roofing asphalt binders using the dynamic shear rheometer (DSR) with parallel-plate geometry. Complex shear modulus can be used to determine complex viscosity (η*)
   4.2. Test specimens, nominally 25-mm in diameter by 1 mm thick or 8-mm in diameter by 2 mm thick, are formed between parallel metal plates.
   4.3. During testing, one of the parallel plates is oscillated with respect to the other at a pre-selected frequency and shear strain. The shear strain is selected so that the testing is expected to be within the linear viscoelastic region of the roofing asphalt binder.
   4.4. The High Temperature Procedure uses a temperature sweep at a fixed frequency to determine the critical temperature (T_c) where the complex shear viscosity, η*, reaches a specified value.
      4.4.1. The T_c value determined by this procedure is a surrogate for the Ring and Ball Softening Point Temperature determined using ASTM D36.
   4.5. The Intermediate Temperature Procedure uses a single point measurement to determine G* at a fixed temperature and frequency
      4.5.1. The value of G* determined by the single point measurement ($G^*_{2.5\ rad/s}$) is a surrogate for the Penetration determined using ASTM D5
   4.6. The Intermediate-Low Temperature Procedure uses a multi-point measurement to determine G* and δ at multiple temperatures (T) and a fixed frequency
      4.6.1. The values of T, G* and δ from the multi-point measurements are used to interpolate the following parameters:

4.6.1.1. δ where G* is 10 MPa ($\delta_{G^*=10\ MPa}$), 10 rad/s
4.6.1.2. Log G* where δ is 27° (log $G^*_{\delta=27°}$), 10 rad/s
4.6.1.3. T where δ is 27° ($T_{\delta=27°}$), 10 rad/s

5. Significance and Use
 5.1. The procedures provide a means of using rheological properties as surrogates for standard tests used to characterize roofing asphalt binders such as Ring-and-Ball Softening Point and Penetration and for characterizing the relaxation properties of an asphalt roofing coating in the high-stiffness region.

6. Applicability and Interferences
 6.1. Refer to ASTM D7175, Section 6 for applicability and notes on potential interferences.

7. Apparatus
 7.1. Refer to ASTM D7175, Section 7 for information on Apparatus.

8. Materials
 8.1. Refer to ASTM D7175, Section 8 for information on Materials.

9. Verification
 9.1. Refer to ASTM D7175, Section 9 for information on Verification of equipment and temperatures.

10. Preparation of Apparatus
 10.1. Refer to ASTM D7175, Section 10 for information on Preparation of Apparatus with the following exceptions
  10.1.1. *Zero Gap (ASTM D7175, Section 10.5)* – For the High Temperature Procedure, zero the gap at 90°C. For the Intermediate Temperature Procedure, zero the gap at 25°C.
  10.1.2. *Preheating 25-mm Plate (ASTM D7175, Section 10.6.1)* – For the High Temperature Procedure, preheat the 25-mm plates to 90°C.
  10.1.3. *Preheating 8-mm Plate (ASTM D7175, Section 10.6.1)* – For the Intermediate Temperature Procedure, preheat the 8-mm plates to 75°C.

11. Preparing Test Specimen
 11.1. *Annealing Roofing Asphalt Binder* – Anneal the roofing asphalt binder sample by heating in a container in an oven until it is sufficiently fluid to pour. Cover the sample and stir it as needed during the heating process to ensure homogeneity and to remove air bubbles. Annealing prior to testing removes reversible molecular associations (steric hardening) that occur during normal storage at ambient temperature. Cold samples must be annealed prior to testing. Structure developed during storage can result in overestimating the modulus by as much as 50%. During annealing process, do not allow the temperature of the roofing asphalt binder to exceed 200C. Always minimize the heating temperature and time to avoid hardening the sample. Hot plates shall not be used to heat the roofing asphalt binder.
 11.2. *Transferring Roofing Asphalt Binder to Test Plate* – Transfer roofing asphalt binder to one of the test plates through pouring, direct transfer, or silicone mold.
  11.2.1. Refer to ASTM D7175, Sections 11.3.1-11.3.3 for information on transferring the binder to the test plates.
 11.3. Trimming Test Specimen

11.3.1. Refer to ASTM D7175, Section 11.4 for information on trimming the test specimen.

11.4. Creating Bulge

11.4.1. Refer to ASTM D7175, Section 11.5 for information on creating the bulge.

12. Test Procedure

12.1. *High Temperature Procedure*

12.1.1. Use 25-mm parallel plate geometry with a 1-mm gap as noted in Section 10.

12.1.2. Load and trim the specimen at 90°C as noted in Sections 10 and 11.

12.1.3. Perform a temperature sweep (90°C, 100°C, 110°C) in oscillatory mode using 5% shear strain and a loading frequency of 1 rad/s.

12.1.3.1. Perform the temperature sweep on the same sample in increasing temperature steps, allowing 10 minutes for thermal equilibrium at each test temperature.

12.1.3.2. Perform the test at each test temperature for twenty loading cycles. Determine the complex shear modulus, G*, in Pa and complex viscosity, η*, in Pa-s as the average of the last ten loading cycles.

Note 1 – Complex viscosity, η*, can be determined by dividing the complex shear modulus, G*, in Pa by the loading frequency in rad/s. With a loading frequency of 1 rad/s, η* in Pa-s is equal in magnitude to G* in Pa.

12.1.4. Unload the sample and clean the DSR.

12.2. *Intermediate Temperature Procedure*

12.2.1. Use 8-mm parallel plate geometry with a 2-mm gap as noted in Section 10.

12.2.2. Set the gap at 25°C. Increase the temperature to 75°C and load, trim the sample. Lower the geometry to the testing gap (2 mm). Reduce the temperature to 25°C and allow the sample to achieve temperature equilibrium before testing.

12.2.3. Start testing at 25°C in oscillatory mode using 0.7% shear strain and a loading frequency of 2.5 rad/s. Perform the test for sixteen loading cycles. Determine the complex shear modulus, G*, in Pa as the average of the last eight loading cycles.

12.2.4. Unload the sample and clean the DSR.

12.3. *Intermediate-Low Temperature Procedure*

12.3.1. Use 8-mm parallel plate geometry with a 2-mm gap as noted in Section 10.

12.3.2. Set the gap at 25°C. Increase the temperature to 75°C and load, trim the sample. Lower the geometry to the testing gap (2 mm).

12.3.3. Perform a temperature sweep in oscillatory mode using 0.1% shear strain and a loading frequency of 10 rad/s.

12.3.3.1. Perform the temperature sweep on the same sample in decreasing temperature steps at intervals of no greater than 6°C, allowing 10 minutes for thermal equilibrium at each test temperature. Test temperatures should be chosen such that G* of 10 MPa (log G* of 7) and δ of 27° will be bracketed.

12.3.3.2. Perform the test at each test temperature for sixteen loading cycles. Determine G* in Pa and δ in degrees as the average of the last eight loading cycles.

12.3.4. Unload the sample and clean the DSR.

13. Calculations

13.1. High Temperature Procedure

13.1.1. Determine the logarithm of the measured complex viscosity (n*) values.

13.1.2. Determine the equation of a best fit line of log n* as a function of temperature (T).

13.1.3. Using the linear regression coefficients, determine the critical temperature, T_c, where n* is equal to 1200 Pa-s (log n* = 3.079) at a frequency of 1 rad/s.

$$T_c = \frac{\log(1200) - B}{A}$$

Where T_c is the critical temperature
A and B are linear regression coefficients of the form log η*= AT + B

13.2. Intermediate-Low Temperature Procedure

13.2.1. Determine the logarithm of the measured complex modulus (G*) values

13.2.2. Determine the equation of the best fit line for log G* as a function of phase angle (δ)

13.2.3. Using the linear regression coefficients, determine the phase angle where log G*=7 at a frequency of 10 rad/s.

$$\delta_{G*=10\,MPa} = \frac{7 - B}{A}$$

Where $\delta_{G*=10\,MPa}$ is the phase angle at which G* is 10 MPa
A and B are linear regression coefficients of the form log G*= Aδ + B

13.2.4. Using the linear regression coefficients, determine the log G* where δ=27° at a frequency of 10 rad/s.

$$\log G^*{}_{\delta=27^\circ} = 27A + B$$

Where $\log G^*_{\delta=27^\circ}$ is the logarithm of the complex modulus at which phase angle is 27 degrees
A and B are linear regression coefficients of the form log G*= Aδ + B

13.2.5. Determine the equation of the best fit line for temperature (T) as a function of phase angle (δ)

13.2.6. Using the linear regression coefficients, determine the temperature where phase angle is 27 degrees at 10 rad/s.

$$T_{\delta=27^\circ} = 27A + B$$

Where $T_{\delta=27^\circ}$ is the temperature at which phase angle is 27 degrees
A and B are linear regression coefficients of the form T= Aδ + B

14. Report

14.1. Sample and Operator information

14.2. Test temperature to indicate the temperature of the test specimen between the DSR plates, in °C to the nearest 0.1°C

14.3. Temperature correction, if a temperature offset was applied, at the test temperature, in °C to the nearest 0.1°C

14.4. High Temperature Procedure – Tc where n* is equal to 1200 Pa-s at a frequency of 1 rad/s, in °C to the nearest 0.1°C

14.5. Intermediate Temperature Procedure – G*, in Pa to three significant figures

14.6. Intermediate-Low Temperature Procedure

14.6.1. $\delta_{G^*=10\ MPa}$, in degrees to the nearest 0.1 degrees

14.6.2. log $G^*_{\delta=27^\circ}$, in Pa to three significant figures

14.6.3. $T_{\delta=27^\circ}$, in °C to the nearest 0.1°C

15. Precision and Bias

15.1. No precision and bias estimates have yet been established for the procedures in Section 12.