U.S. Department
of Transportation
**Federal Highway
Administration**

Publication No. FHWA-SA-94-068

July 1994

SUPERPAVE ASPHALT BINDER TEST METHODS

AN ILLUSTRATED OVERVIEW

NATIONAL ASPHALT TRAINING CENTER DEMONSTRATION PROJECT 101

Asphalt Institute

TECHNOLOGY APPLICATIONS
Innovation Through Partnerships

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Technical Report Documentation Page

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
FHWA-SA-94-068		

4. Title and Subtitle	5. Report Date
SUPERPAVE Asphalt Binder Test Methods An Illustrated Overview	January 1994
	6. Performing Organization Code
7. Author(s) R.S. Warren, R. B. McGennis, H.U. Bahia	8. Performing Organization Report No.
9. Performing Organization Name and Address Asphalt Institute P.O. Box 14052 Lexington, KY 40512-4052	10. Work Unit No. (TRAIS)
	11. Contract or Grant No. DTFH61-92-C-00098
12. Sponsoring Agency Name and Address Federal Highway Administration Office of Technology Applications 400 Seventh St., SW Washington, DC 20590	13. Type of Report and Period Covered Final Report December 1992-January 1993
	14. Sponsoring Agency Code HTA-21

15. Supplementary Notes

FHWA Technical Representative - J.R. Bukowski

16. Abstract

This manual provides sequential, illustrated steps for performing the SUPERPAVE test procedures on asphalt cements. It also serves as a self-contained laboratory reference document on those procedures. These tests and procedures represent the results of the SHRP 5 -year research effort to investigate and improve asphalt cement technology. This manual was developed under the FHWA's National Asphalt Training Center

17. Key Words	18. Distribution Statement
Asphalt cement, binder, dynamic shear rheometer, bending beam rheometer, pressure aging vessel, direct tension devices, PG grading system	No restrictions. This document is available to the public from the National Technical Information Service, Springfield, Virginia 22161

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price
Unclassified	Unclassified	140	

Form DOT F 1700.7 (8-72) Reproduction of completed page authorized

FOREWORD

From October 1987 through March 1993, the Strategic Highway Research Program (SHRP) conducted a $50 million research effort to develop new ways to test and specify asphalt binders. Near the end of SHRP, the Federal Highway Administration assumed a leadership role in the implementation of SHRP research. An integral part of FHWA's implementation strategy was a project to develop a nationally accessible training center aimed at educating both agency and industry personnel in the proper use and application of the final SHRP asphalt products referred to as SUPERPAVE™. This project was administered by the FHWA's Office of Technology Applications and designated Demonstration Project 101, the National Asphalt Training Center (NATC).

The NATC resides at the Asphalt Institute's Research Center in Lexington, Kentucky. While the day-to-day affairs of the NATC are directed by Institute personnel, course development and technical direction were duties shared by a team of engineers and technologists from the Asphalt Institute, the Pennsylvania State University, the University of Texas at Austin, the National Center for Asphalt Technology, Marathon Oil Company, and FHWA.

The principal objective of the educational program is to train students in the proper use of the new SUPERPAVE binder test methods and equipment. Another key objective is to teach students how to interpret and apply the new SUPERPAVE binder specification. The training program consists of 40 hours of instruction. Of this 40 hours, students receive eight hours of classroom instruction, 28 hours of laboratory instruction, and four hours of classroom discussion of actual test results. By the end of the course, students will be familiar with binder test procedures and equipment and will know how to use binder test results to classify binders according to the SUPERPAVE binder specification.

The purpose of this manual is to provide a laboratory instructional reference that will be used by students at the Center as they are instructed in asphalt binder test procedures. This instruction occurs principally in the NATC laboratories. The manual is written for technicians and engineers with no previous training in SUPERPAVE products, but with some knowledge in asphalt technology. Other instructional aids at the Center include a student text that is used in the classroom portion of training.

Included in Appendix A of this manual is a set of AASHTO Provisional Standards (September 1993) for Asphalt Binders and other current AASHTO procedures. This manual provides an illustrated overview of these test methods but it is not intended to replace them. The AASHTO Provisional Standards provide detailed information pertaining to all aspects of the tests and form the most important tool in properly performing binder testing.

The illustrated procedures contained in this manual should be used with great caution. It was developed during the last stages of SHRP and in the first few months following SHRP's completion. While the test procedures were largely complete by this time,

specific details of the procedures were (and still are) changing. During preparation of this illustrated overview, no fewer than four drafts of the test methods were available. The steps outlined in this manual follow, as closely as possible, the procedures outlined in the standards contained in Appendix A. *Users of the manual are strongly encouraged obtain the latest versions of the AASHTO test procedures as the most up-to-date reference.*

One of the principal benefits of many of the new SUPERPAVE binder test procedures is their reliance on computer control and data acquisition. The dynamic shear rheometer, bending beam rheometer, direct tension tester, and some rotational viscometers use computers to control testing and capture test results. Because this manual was prepared for use at the National Asphalt Training Center, it is written around the testing equipment and related computer software used for training at the Center. Users should be aware that future developments in binder test equipment and software may conflict with information in this manual. In such cases, users should implicitly follow operational guides and software instructions from the equipment manufacturers along with the most current AASHTO standards.

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January 1994
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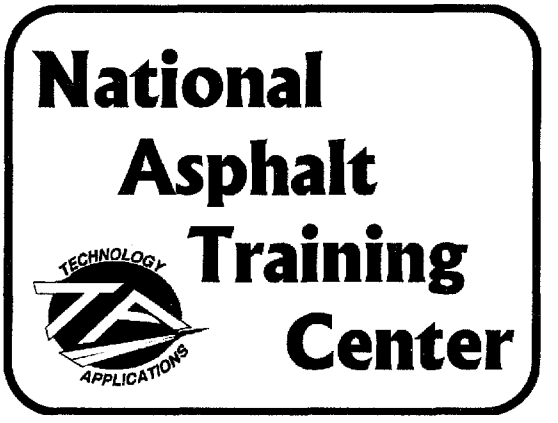


Dynamic Shear Rheometer

Test Method for Complex Modulus (G*) and Phase Angle (δ) of Asphalt Binders Using a Dynamic Shear Rheometer

This document outlines the basic steps in measuring the complex shear modulus (G*) and the phase angle (δ) of asphalt binders according to AASHTO Provisional Method TP5. It provides an illustrated overview, but it is not intended to replace the standard test method which contains detailed information pertaining to all aspects of the test.

1. This procedure outlines determination o' complex shear modulus and phase angl of asphalt binders. The apparatus consists of a dynamic shear rheometer (DSR), a temperature controller, and a computer that is used to control the rheometer and acquire data. The test is performed on unaged binders and binders that have been aged in a rolling thin film oven and pressure aging vessel. One apparatus is shown in Figure 1.

2. At the beginning of the procedure, a 10 g sample is usually in a small container such as a "3 ounce tin." To prepare for testing heat the sample until it is sufficiently fluid to pour. The consistency should be less than 0.5 Pa-sec which is the approximate consistency of motor oil. The sample should never be heated above 150° C.

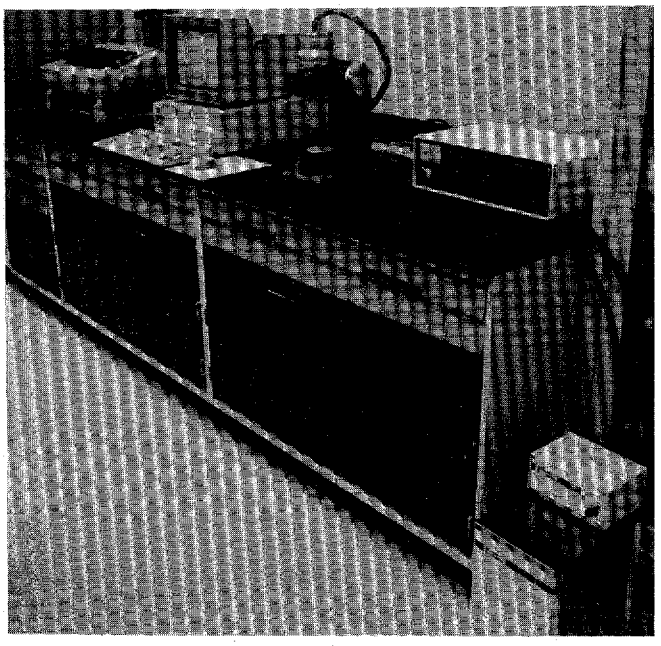


Figure 1. DSR Apparatus

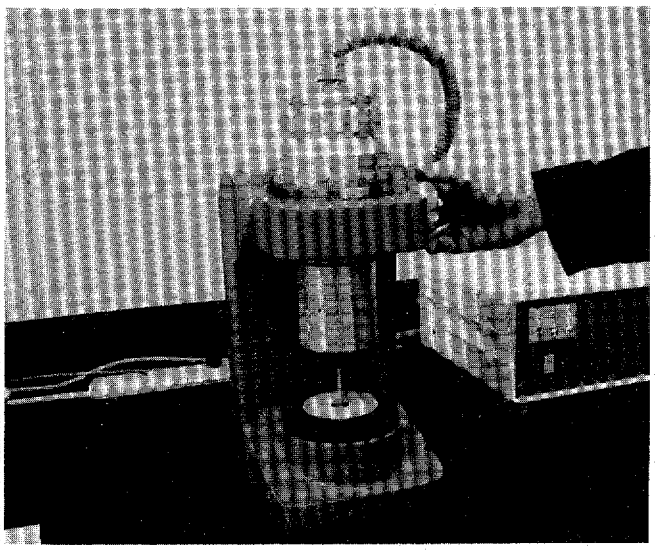


Figure 2. Dynamic Shear Rheometer

3. Two acceptable techniques can be used to procure a specimen for testing. One technique uses a small silicone rubber mold to fabricate a test specimen. The other technique simply requires the operator to pour close to the required amount of asphalt directly onto the upper or lower plate (Figure 3). This method is fast and simple but requires experience in pouring the precise amount needed for testing. The silicone rubber mold procedure is slightly more time consuming, but since it results in a more precise sample size, requires less trimming.

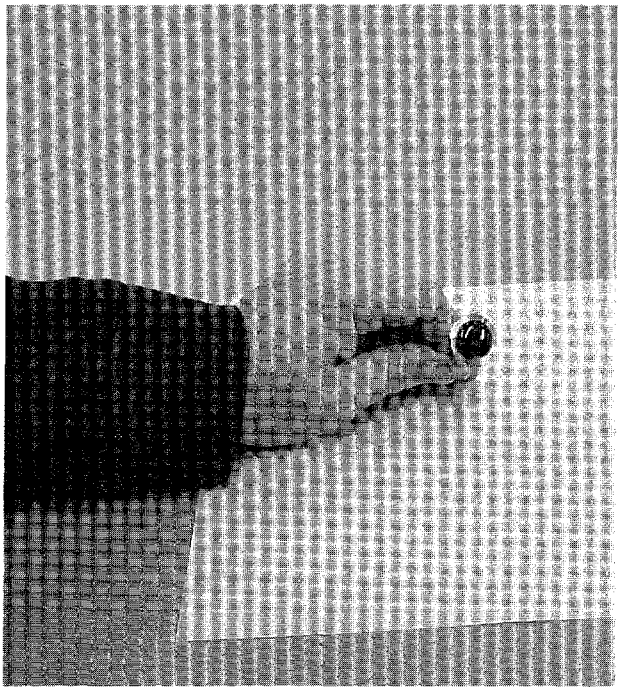


Figure 3. Asphalt Applied to Upper Plate

4. To use the silicone rubber mold procedure, pour a thin stream into the mold center (Figure 4) and allowing it to cool at room temperature for about 10 minutes. While most samples can be demolded (Figure 5) at room temperature, some soft samples require slight chilling in a freezer for 5 minutes or less prior to demolding. The sample would be applied directly to the plate before testing and not handled.

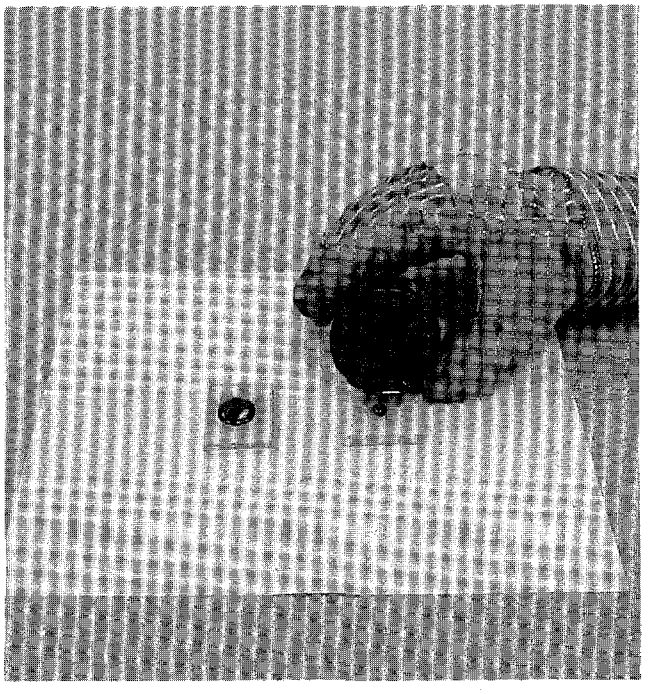


Figure 4. Pouring Hot Asphalt Into Mold

5. While the sample is being prepared, the operator should prepare the rheometer for testing.

6. Turn on the rheometer air system by opening the supply regulator. The regulator is a valve affixed to the central laboratory air system and is normally located close to the rheometer. In many cases, the valve is part of a combination regulator/water filter system and is not part of the rheometer itself. It is important that the rheometer air system be on prior to manipulation of the rheometer to prevent damage to any components. Turn on personal computer system and temperature control system. Figure 6 is one type of temperature control system that circulates water.

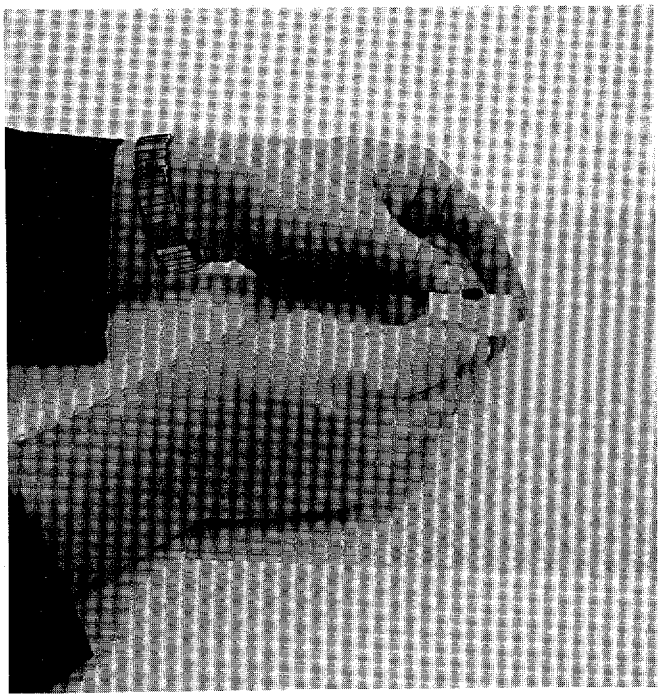


Figure 5. Flexing Mold to Remove Specimen (specimen would be applied directly to plate)

7. Turn on rheometer. Refer to the operator's manual for detailed instructions, such as location of the on/off switch, etc.

8. Initialize rheometer software program. The main menu will appear on computer screen. If rheometer is equipped with a calibration or equipment check routine, perform this step now. Different brands of rheometers require varying degrees of such checks. Consult the operator's manual for details.

9. From the main menu, select or verify that the rheometer is in the oscillatory load mode.

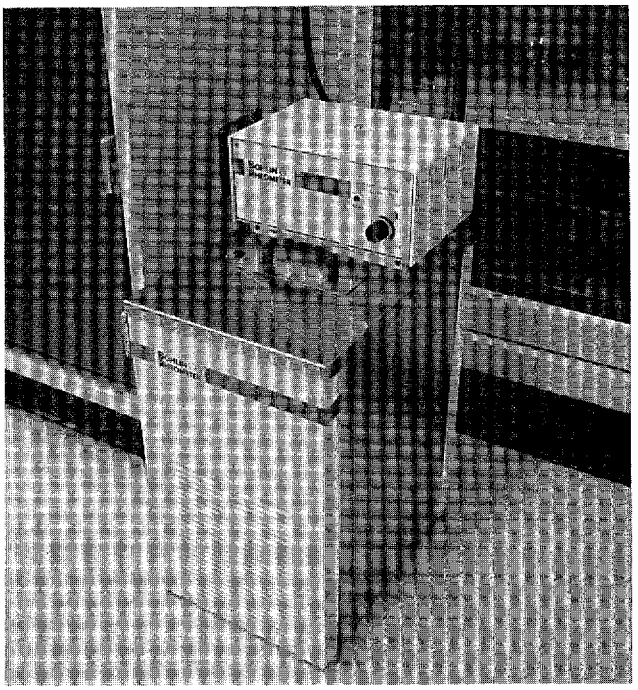


Figure 6. Temperature Control System

10. Use the rheometer software to set the proper base plate test temperature. If only one test temperature is to be used, set the rheometer accordingly. If using a range of temperatures, set the base plate temperature in the middle of this range.

11. When the computer screen indicates the desired temperature has been reached the operator can set the gap between the upper plate and base plate. This is accomplished in two distinct steps: establishing the zero gap position and the desired gap position.

12. Attach the upper plate to the rheometer spindle. Two plate sizes are used. A small plate, 8 mm in diameter, is used to perform tests at moderate temperatures of about 40° C or below. A large plate, 25 mm in diameter, is used to perform tests at higher temperatures greater than 46° C. Figure 7 shows various sizes of plates.

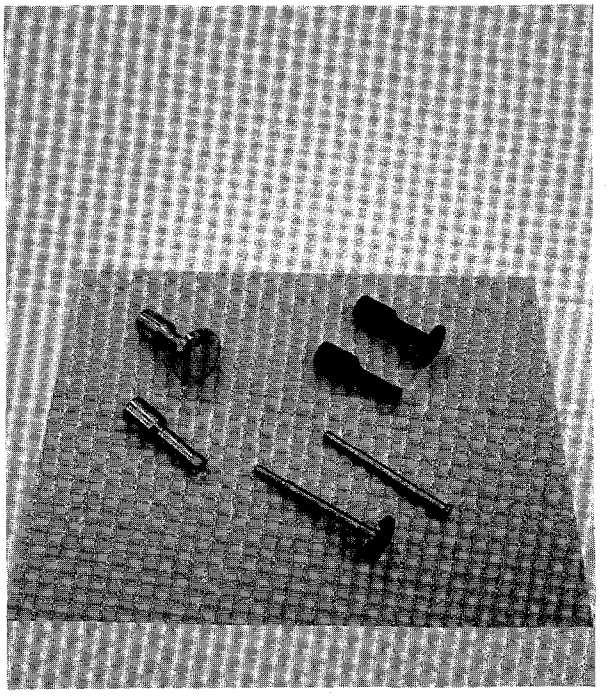


Figure 7. Various DSR Plates

13. Depending on the type of rheometer, either lower the upper plate or raise the base plate (Figure 8) so that they are in close proximity (approximately 3 mm). These large adjustments are made using a variety of techniques, which are rheometer dependent. In some cases, the upper plate is lowered with a coarse adjustment knob similar to a drill press (Figure 2). In other cases, the lower plate is raised by means of a hydraulic ram (Figure 9). The operator should consult the rheometer instruction manual to determine how to adjust the gap.

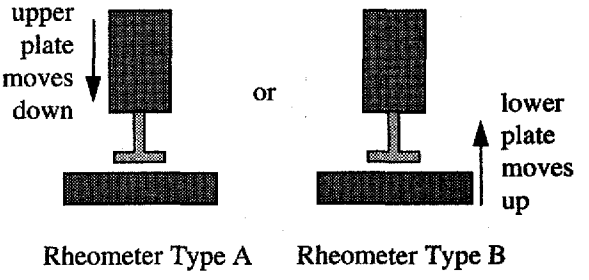


Figure 8. Methods of Adjusting Gap Between Upper and Lower Plates

14. Gently spin the spindle and upper plate. The point where the upper plate contacts the base plate and just stops spinning is considered a zero gap condition. After zero gap position has been established, the operator should read and record the number off the micrometer wheel. The micrometer wheel indicator is usually located above the upper plate or below the base plate and functions as a vertical position indicator to establish the position of the base plate and upper plate relative to each other. In Figure 9, the micrometer wheel is in the small oval window in front of the ram. Figure 10 shows the micrometer wheel on another rheometer. Graduations on the micrometer wheel are normally in 5 micron increments; the operator should consult the operator's manual if questions arise over this scale. This procedure of spinning the upper plate followed by gentle contacting with the base plate to establish zero gap should be performed several times to firmly establish that a zero gap has been achieved. When two successive micrometer wheel readings are 2 microns or less apart, the operator can be assured that a zero gap condition has been achieved. It should be noted that the zero gap condition assumes that the upper plate and base plate are barely in contact, and hence, the upper plate is no longer free to spin. On some rheometers, the micrometer wheel indicator can be reset to read "zero" when the zero gap position has been achieved. For other rheometers without this capability, it is sufficient to simply record the observed number on the micrometer wheel when the rheometer is in the zero gap position to establish the base or "zero" reading.

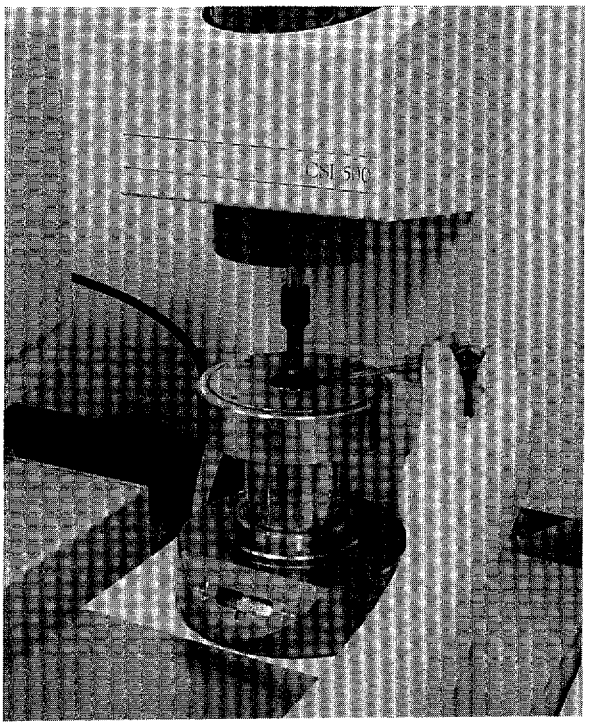


Figure 9. Hydraulically Activated Lower Plate (Note specimen being trimmed)

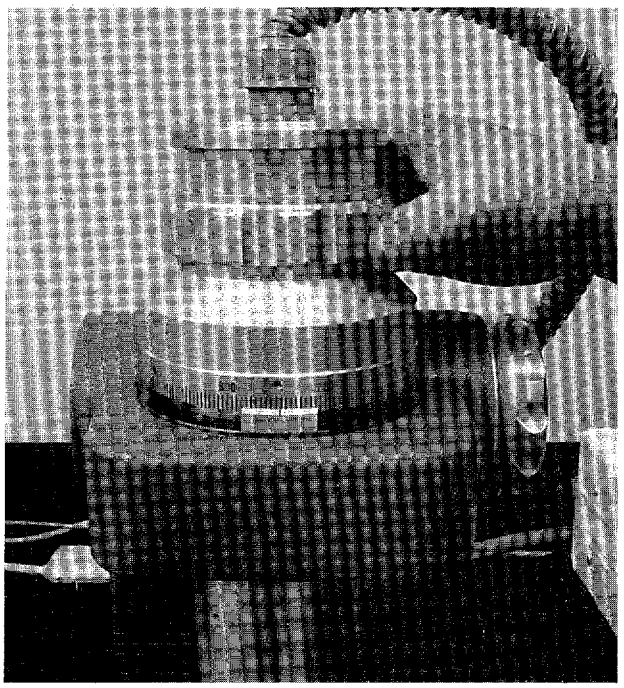


Figure 10. Micrometer Wheel

15. Once the operator sets the zero gap condition, the testing gap can be set. For the larger plate used at high test temperatures of 46° C or more, the gap between the upper plate and base plate should be set at 1 mm. The smaller plate used at intermediate test temperatures of 40° C and below require a gap of 2 mm.

16. When initially setting the gap between the upper plate and base plate, the operator sets a gap equal to the desired gap plus an extra 50 microns (Figure 11). The extra 50 microns will be dialed-out prior to testing but is included to achieve the proper sample shape during testing.

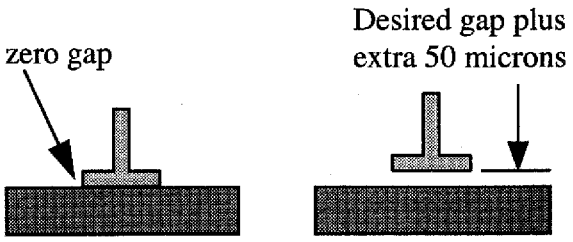


Figure 11. Extra Gap Set by Operator

17. Set the desired gap (including the extra 50 microns) by dialing the micrometer wheel to 1 mm plus 50 microns (high test temperature gap) or 2 mm plus 50 microns (low test temperature gap). Normally, one full revolution of the micrometer wheel results in a 1 mm (1000 microns) change in vertical position.

18. A skilled operator should be able to prepare the rheometer for testing, including all gap settings in 5 minutes or less.

19. The operator should clean and dry the upper and lower plates before testing. Acetone is a suitable fluid for cleaning and drying.

20. The exact steps in installing the binder sample are rheometer dependent. The two most common procedures are as follows.

21. For rheometers that have an activated base plate such as shown in Figure 12, lower the base plate to its lowest vertical position. This is normally accomplished by using the rheometer control software. Pour the heated sample onto the base plate. Pour the sample in a single, thin stream so that it spreads to form a disc that has a diameter within about 2 mm of the upper plate. If a molded sample is used, flex the silicone mold (Figure 5) and apply the molded sample directly to the base plate. The operator should not use fingers to handle or position the molded sample. A clean tool may be used for this purpose. Use the rheometer software to raise the base plate back up to its previous position, at which the preliminary gap had been set. This gap setting should not have changed. The sample should now be squeezed between the upper and base plates, ready for trimming (Figure 12).

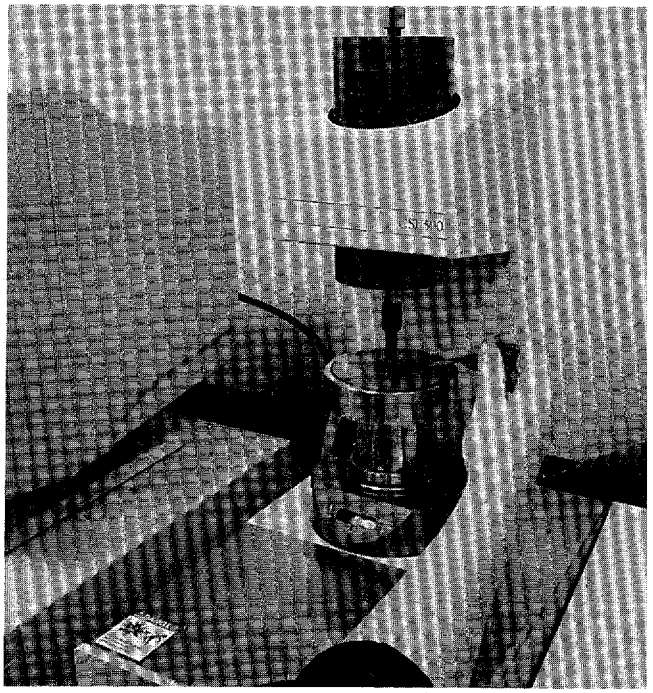


Figure 12. Base Plate Raised Squeezing Sample in Gap; Sample Being Trimmed

22. For rheometers with an activated upper plate, the sample is installed on the upper plate. Thus, the first step is to raise the upper plate (Figure 13) and remove it from the rheometer. Invert the plate and pour the sample in a single, thin stream (Figure 14) so that it spreads to form a disc shape that has a diameter within about 2 mm of the upper plate. If a molded sample is used, flex the silicone mold and apply the molded sample directly to the upper plate (Figure 5). The operator should not use fingers to handle or position the molded sample. A clean tool may be used for this purpose. Reinstall the upper plate. It now has the cooled sample stuck to its surface. Lower the upper plate back down to its previous position, at which the preliminary gap had been set. This gap setting should not have changed. The sample should now be squeezed between the upper and base plates, ready for trimming.

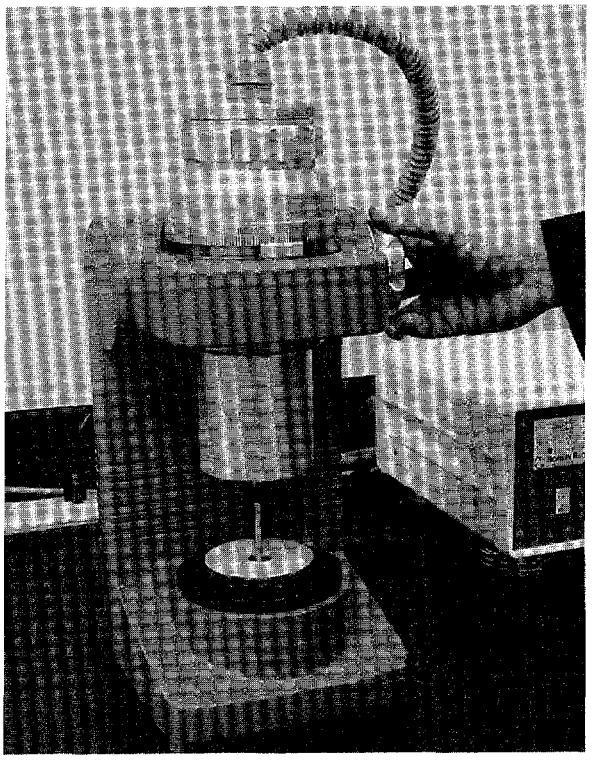


Figure 13. Adjusting Position of Upper Plate

23. Trim the sample using an appropriate, heated tool. Such a tool should be in the shape of a small slotted screwdriver with a slot width greater than 4 mm. Trimming should result in a specimen that has vertical sides, flush with the edge of the upper plate.

Figure 14. Applying Specimen to Upper Plate

24. Next, the operator should dial out the extra 50 microns by adjusting the micrometer wheel. At this point, the gap should be exactly the desired testing gap, either 1 or 2 mm, and the sides of the specimen should bulge slightly (Figure 15). The specimen is now ready for thermal conditioning.

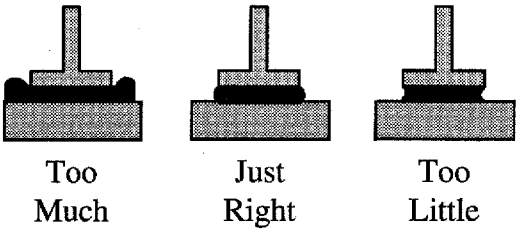


Figure 15. Proper and Improper Sample Shapes

25. Because the properties of asphalt binders are extremely sensitive to temperature, it is necessary to precisely control the temperature of the specimen during the test. The temperature of the sample must be within ±0.1° C of the desired test temperature. Sample temperature is controlled by surrounding it with a circulating water bath (Figure 16) or air oven (Figure 17) that is precisely maintained at the testing temperature. Typically, it takes 10 minutes for the sample to achieve the desired test temperature when an air oven is used. Water baths normally take less time, usually about 5 minutes. The actual amount of time can be determined using a dummy specimen described in step 26.

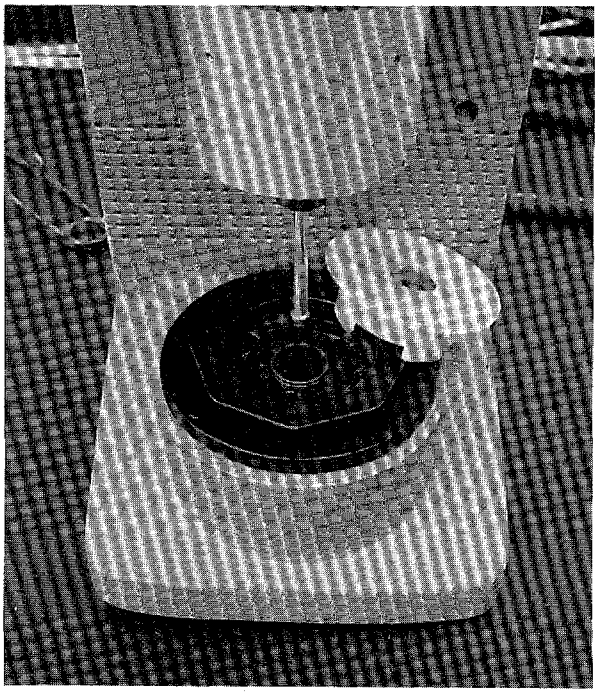


Figure 16. Water Bath for Controlling Specimen Temperature During Testing

26. To ascertain the temperature control characteristics of rheometers, a dummy specimen may be used. One example is the thermistor unit shown in Figure 18. The thermistor unit consists of a resistor embedded between two sheets of silicone rubber. The resistance of the resistor is highly temperature dependent. A calibration is used to convert a change in resistance to a corresponding change in temperature. The unit is placed between the upper and lower rheometer plates and connected to an ohmmeter. Thus, a highly accurate determination of temperature between the plates can be made. By using this approach, the operator can learn the proper amount of time for binder specimens to reach the desired test temperatures. This step need not be performed before each test but rather, on a periodic basis to calibrate the rheometer temperature control system and determine equilibration time (step 27).

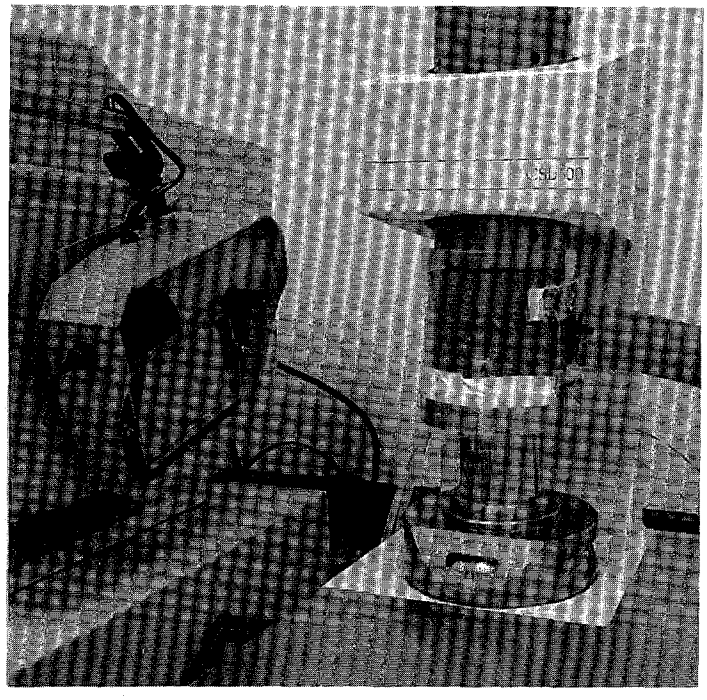


Figure 17. Air Oven for Controlling Specimen Temperature During Test

27. After the temperature reading has reached the desired value, allow the temperature to equilibrate. The equilibration time will be different for different rheometers, temperature control systems, and starting temperature. The equilibration time can be determined using the dummy specimen procedure outlined in step 26. After the temperature has equilibrated, anneal the specimen for 5 minutes (water bath) or 10 minutes (air oven).

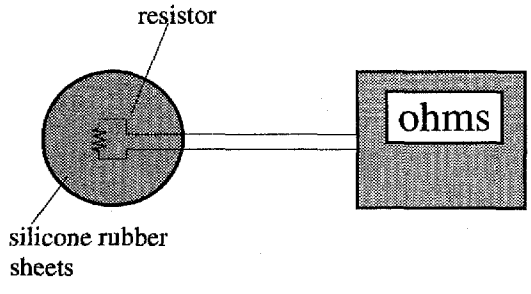


Figure 18. Thermistor Unit Used to Measure Temperature Between Plates

28. At this point the sample is ready to be tested and the operator should use the rheometer software to set the desired test inputs and conduct the test (Figure 19). Once again, the operator should consult the rheometer manual if questions arise concerning the exact steps necessary to perform testing. In most cases, system software is sufficiently self explanatory and often prompts the operator for needed items. Software often contains default values for items such as gap, frequency, etc. and the operator need not worry about setting these values. In other cases, the operator sets inputs the first time and the software "remembers" these values for subsequent tests. The following testing procedure consists of steps that are generic to most rheometers.

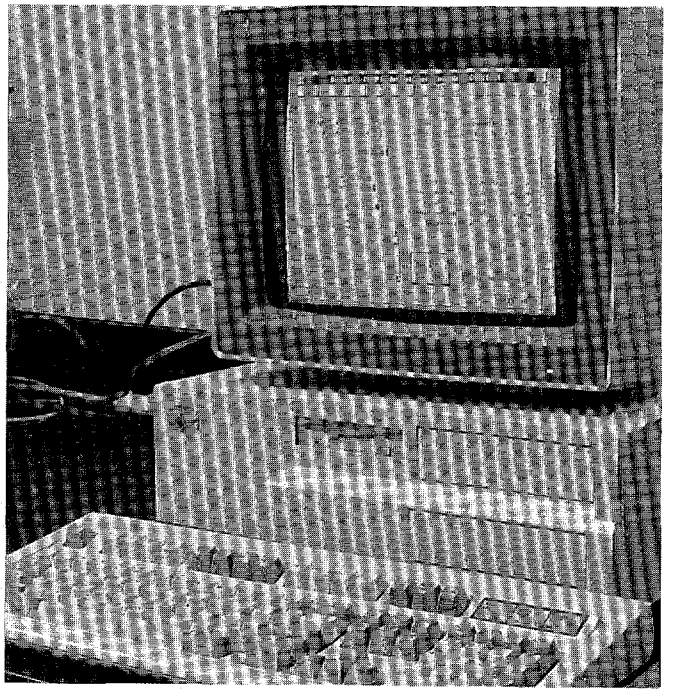


Figure 19. Computer for Controlling Test

29. Using the system software set the desired inputs or verify that the software has selected the proper values. The following items are of interest.

30. Most control software requires the operator to input items like sample identification number, sample age condition, and operator name.

31. The frequency always should be set at 10 radians per second (1.59 Hz).

32. Verify that the gap displayed on the computer screen is the proper value. By so doing, the operator is not resetting the gap between the upper and base plates. This displayed value is simply that which is used by the software to perform subsequent calculations.

33. The displayed plate size should be set or verified by the operator. This value will be either 8 or 25 mm, depending on test conditions.

34. The shear strain or shear stress should be set by the operator. The proper testing values depend on the value of the complex modulus (G*), but the operator can use the values shown in the table (right) as a starting point. After setting a value, the rheometer software will automatically control the shear strain or shear stress during the test.

Material Tested	Shear Strain, %	Shear Stress, kPa
Orig Binder	12	0.12
RTFO Residue	10	0.22
PAV Residue	1	50

35. Initiation of the test may occur in several different ways. Some rheometers will automatically begin testing after the specified temperature equilibration time has been reached. Other rheometers require a manual initiation by the operator. In any case, the operator should follow the directions given by the control software and /or the operator's manual.

36. A test consists of two phases, load conditioning and data acquisition. The conditioning phase consists of a complete run time application of load cycles. During this phase, the sample is subjected to the same shear loads but no tests results are captured. In the data acquisition phase, the sample is similarly loaded and system software captures the response of the sample to the load application and computes test results. During testing, the operator may watch the computer screen and observe the acquisition of test results.

37. Condition the specimen by applying the required shear strain (or stress) for 10 cycles at the required rate of 10 radians per second. Obtain test measurements by loading the specimen for an additional 10 cycles. Test results are automatically acquired by the rheometer software.

38. At the end of the testing sequence, the operator should manipulate the software to get a printed copy of test results (Figure 20) and if necessary, store results on the computer's hard disk drive.

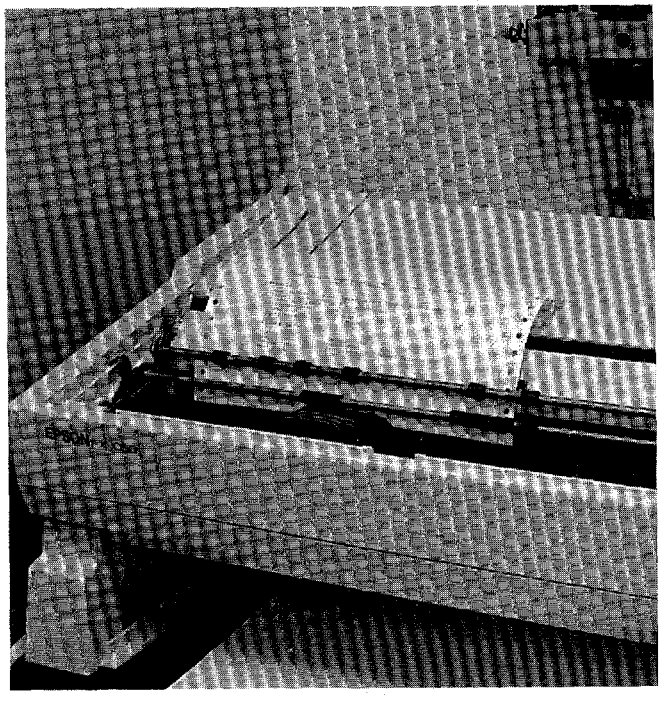


Figure 20. Printer for Printing Test Results

39. All of the required reporting items are printed by the rheometer software. These are:

- test plate size, nearest 0.1 mm,
- test gap, nearest μm,
- test temperature, nearest 0.1° C,
- test frequency, nearest 0.1 rad/sec,
- strain amplitude, nearest 0.01%, or torque, nearest mN-m,
- complex modulus (G*) for the second 10 cycles, kPa to three significant figures, and,
- the phase angle (δ), nearest 0.1°.

Additional reporting items include a complete identification and description of the material tested, including name code, source, and type of sample container. The instrument used for the test should also be described, including whether it is a constant strain or stress device, the type of environmental chamber, and any other information to describe the rheometer.

40. The operator may wish to run additional tests on the same sample, but at a different temperature. In these cases, all that need be done is to reset the temperature control to the desired temperature and allow sufficient time for the specimen to reach the desired temperature followed by temperature equilibration and annealing. The test is performed as before. When multiple temperatures are to be used, the operator should start at a midrange temperature and proceed to next desired temperature.

41. Total time, including sample and equipment preparation, to perform this procedure should be about 1 hour for a skilled operator. This time will increase if test results at different temperatures are required. Each additional testing temperature will normally add about 20 minutes to total test time. When testing one sample at multiple temperatures, the entire testing must be completed within 4 hours from the time the sample was heated to pouring temperature.

42. The operator should clean the upper plate by heating it (if necessary) and wiping with mineral spirits or other suitable solvent. The warm base plate should be wiped with a paper towel to remove excessive binder. The remaining residue should be removed by wiping the base plate with a paper towel moistened with solvent. Always use acetone to clean the plates to remove solvent residue.

43. Rheometer calibration and verification should be performed periodically. This includes calibration of the resistance thermal detector (RTD), load transducer, and strain transducer. A confidence check on the rheometer can be performed using suitable reference fluids or a dummy specimen with known properties. Calibration procedures are provided in the test method.

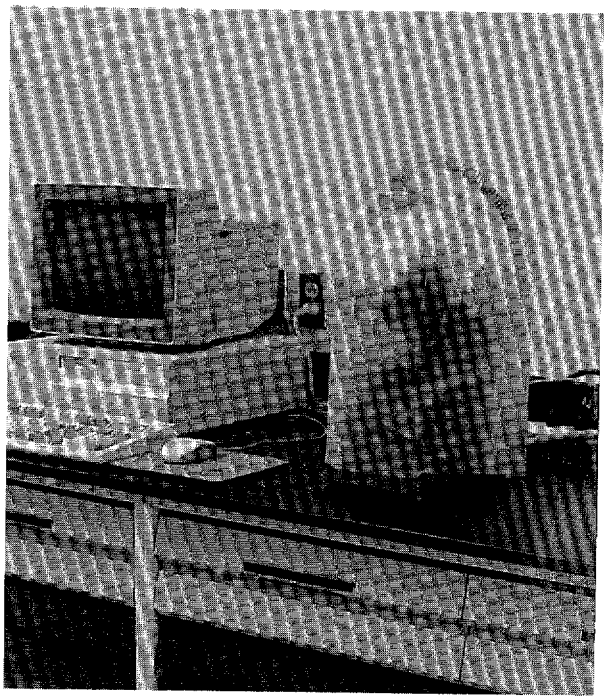
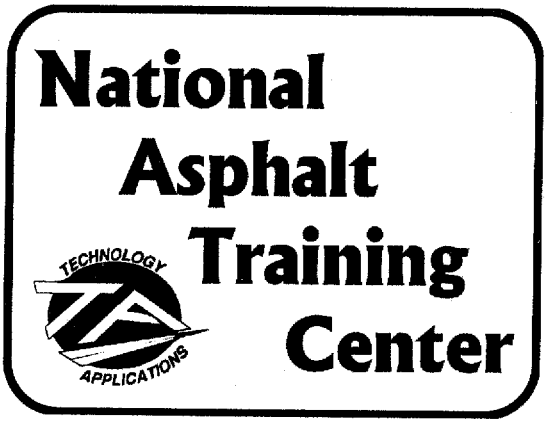


Figure 21. Dynamic Shear Rheometer



Rotational Viscometer

Test Method for Measuring Viscosity of Asphalt Binders Using a Rotational Coaxial Cylinder Viscometer

This document outlines the basic steps in measuring the viscosity of asphalt binders using a rotational coaxial cylinder viscometer according to procedures outlined by ASTM D 4402. It provides an illustrated overview, but it is not intended to replace the standard test method which contains detailed information pertaining to all aspects of the test. This procedure assumes that the operator is using a Brookfield (or similar) Viscometer (DV-II+) with a Thermosel™ temperature control system.

1. This procedure outlines determination of high temperature viscosity of asphalt binders. The apparatus consists of a rotational coaxial cylinder viscometer (Figure 1) and a unit to control temperature (Figure 2). The test is performed on unaged binders.

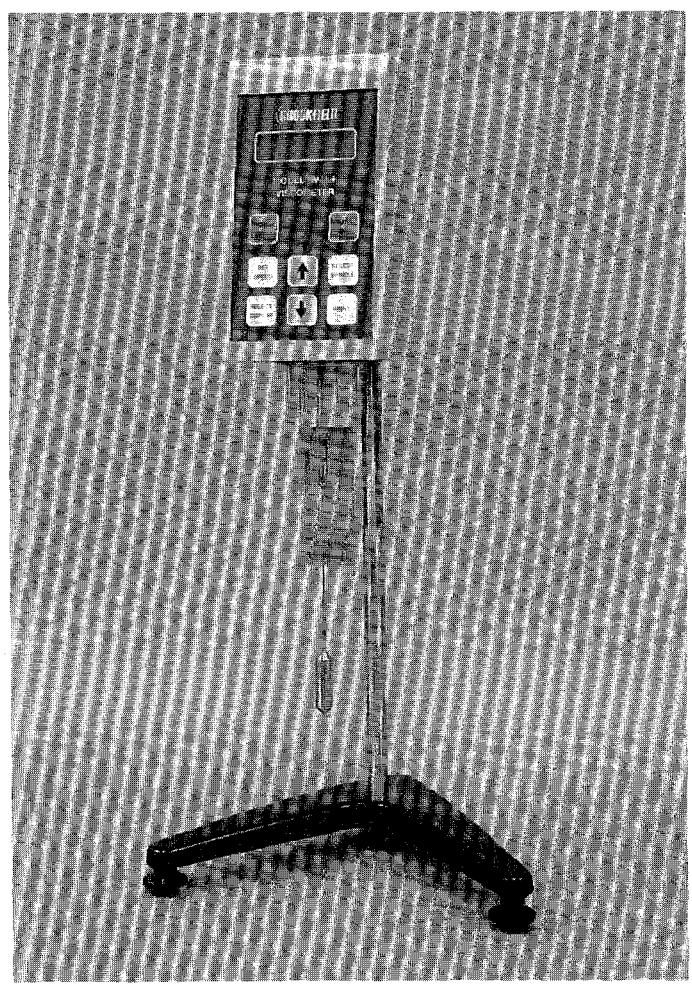


Figure 1. Rotational Viscometer with Spindle Attached

2. Turn on the Thermosel™ (Figure 2). Set the desired test temperature, usually 135 ° C, on the Thermosel controller by holding the set button and turning the set point knob until the digital display reads the proper temperature.

3. Place the sample chamber into the sample chamber holder and then both into an oven at 135° C. In the same oven, place a "3-ounce" container containing the asphalt sample. Also place the spindle in the oven.

4. Turn on the viscometer and remove spindle if necessary. Level the viscometer and thermo container using the bubble indicators on each device.

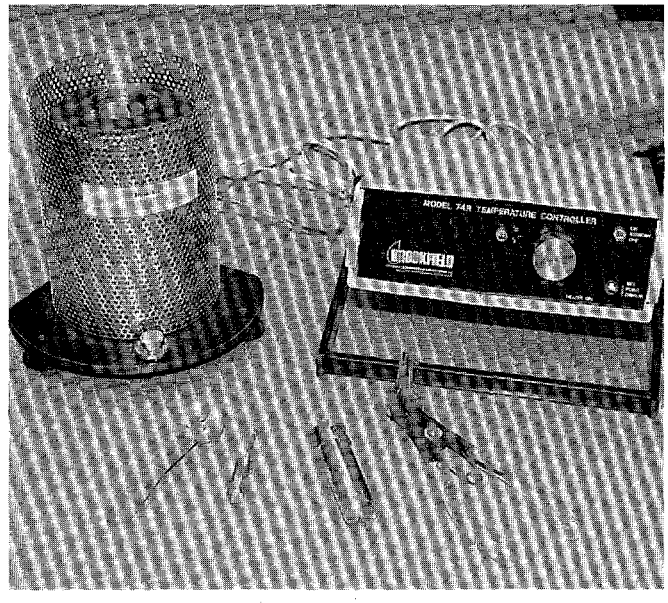


Figure 2. Temperature Control System (background is thermo-container and controller; foreground is insulating cap, spindle, sample chamber, and extraction tool)

5. By using the keypad on the front of the viscometer (Figure 3), push the "Select Spindle" button and press arrow keys until the proper spindle number is displayed. For most unmodified asphalts, spindle nos. 21 and 27 are used. For soft asphalts or modified asphalts, consult the Thermosel system operator's manual for the proper spindle. The spindle used is determined by the anticipated viscosity of the fluid being tested.

Figure 3. Viscometer Keypad

6. When the digital display on the thermosel indicates the desired temperature has been reached, remove the sample chamber still in its holder from the oven and place both on a scale accurate to the nearest 0.1 g. Tare the scale.

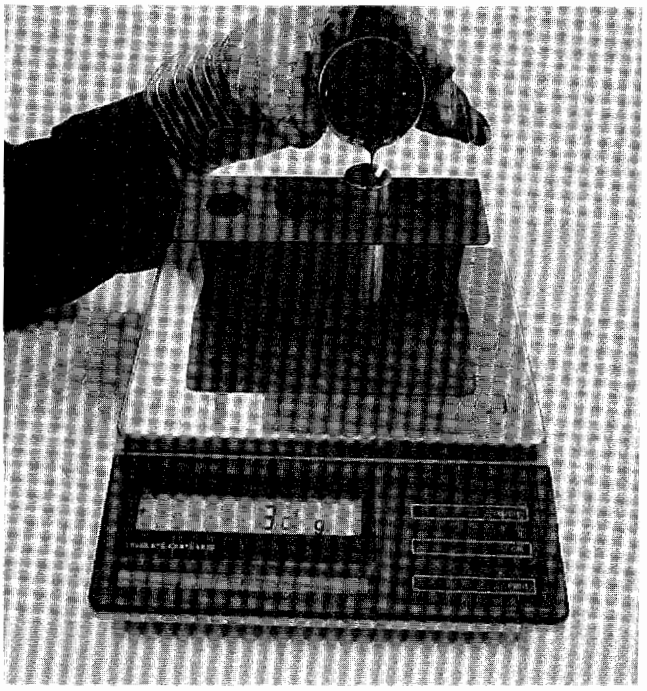


Figure 4. Pouring Sample Into Chamber

7. Pour the required amount of asphalt binder in to the sample chamber (Figure 4). The amount of asphalt used depends on the spindle size. For spindle no. 27, 10.5 ml of asphalt is the correct amount to be loaded into the sample chamber. Consult the operator's manual for the proper sample size for other spindles.

8. Using the extraction tool, remove the sample chamber containing the hot sample from the chamber holder and place it in the thermo-container (Figure 5).

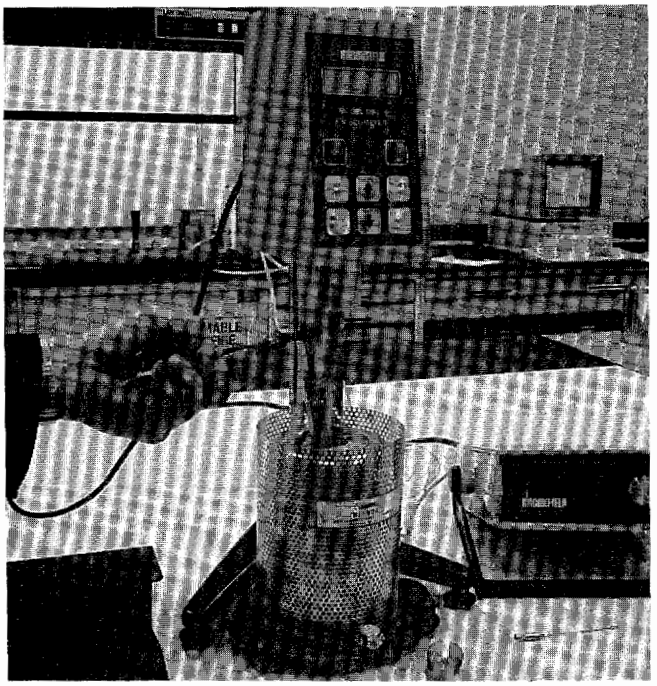


Figure 5. Placing Sample Chamber in Thermo-Container

9. Align the thermo container with the viscometer. To accomplish this, lower the viscometer by turning the height adjustment knob until the alignment bracket makes contact with the rear vertical face and horizontal face of the locating ring on the thermo-container (Figure 6). From this position, raise the viscometer approximately 1.5 mm. Since this is such a critical measurement, a reference point should be marked on the rear vertical face of locating ring to facilitate precise vertical positioning.

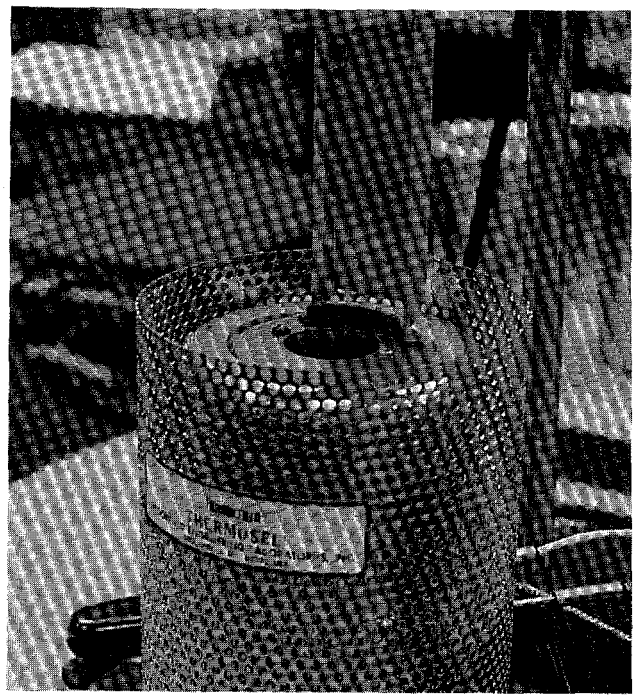


Figure 6. Alignment Bracket and Locating Ring

10. Remove the spindle from the oven and attach it to the spindle extension (Figure 7). The spindle extension is a stiff wire with a loop on one end and a female coupling on the other. To attach the spindle, simply insert the loop through the small hole in the top of the spindle.

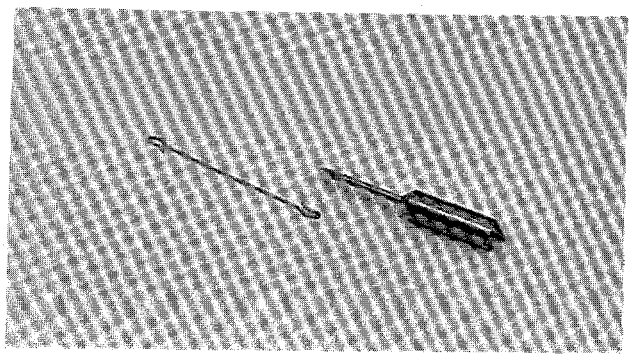


Figure 7. Spindle and Extension

11. Gently lower the spindle into the sample chamber containing the hot sample. Lower it enough so that the female coupling on the end of the spindle extension can be screwed onto the male viscometer coupling nut (Figure 8).

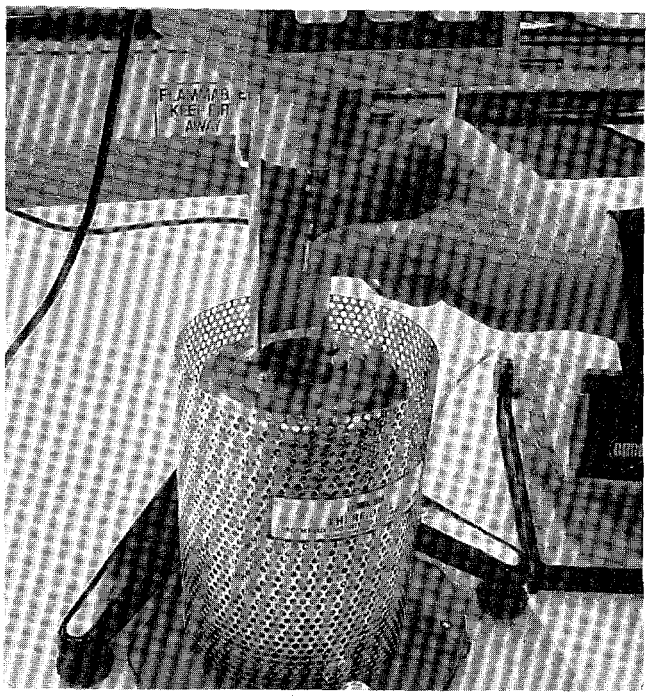


Figure 8. Attaching Spindle Extension with Viscometer Coupling Nut

12. Place the insulating cap over the opening in the thermo container (Figure 9). Equilibrate the sample temperature for a period of approximately 30 minutes. During this period, occasionally observe the digital temperature display on the controller to verify that the temperature is rising toward the test temperature.

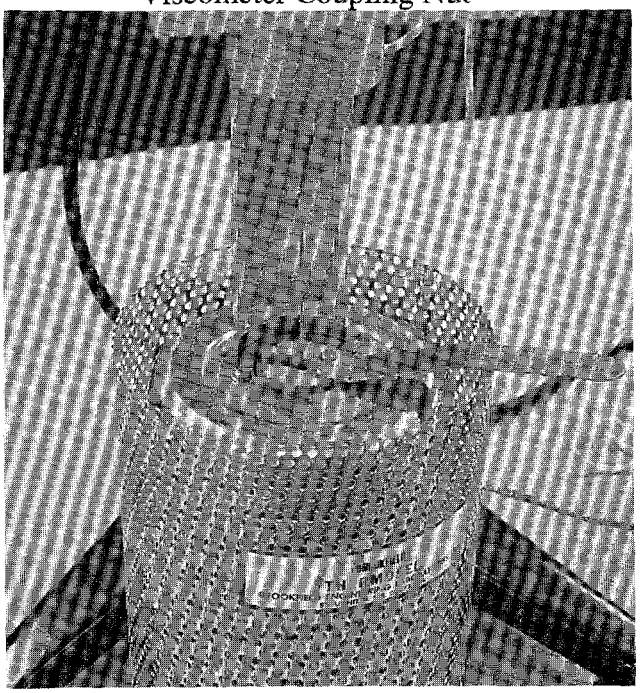


Figure 9. Insulating Cap on Thermo-Container

13. Set the viscometer motor speed by pressing the "Speed" key on the viscometer keypad (Figure 10). Use the arrow keys to set the desired testing speed, 20 rpm. For relatively soft binders, the speed may need to be increased in order to increase the viscometer torque value so that it is within the acceptable range of 2 to 98 percent torque.

14. Set the display to read viscosity by pressing the "Set Display" key until viscosity in centipoises (cP) is shown in the upper left corner of the display. During the equilibration period, observe the viscosity. The viscosity will normally decrease as the temperature of the sample rises. When the viscosity reading remains constant, the temperature is considered equilibrated.

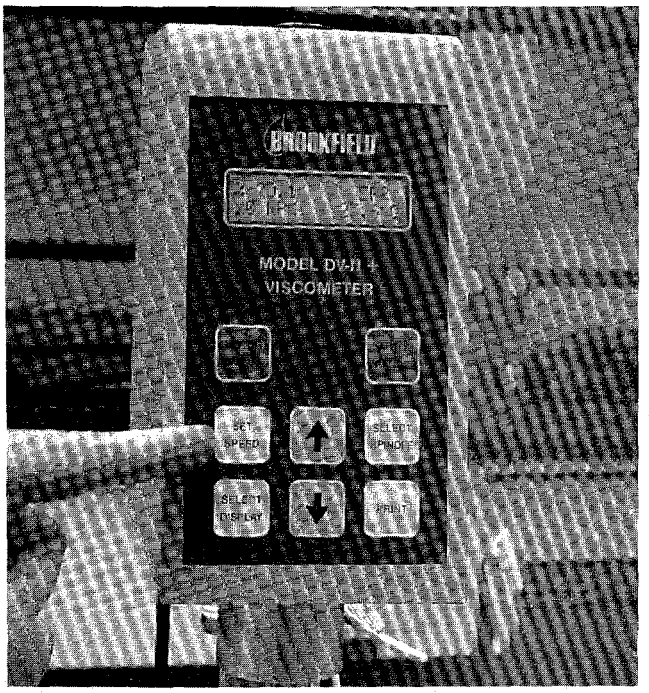


Figure 10. Viscometer Keypad

15. Read and record a viscosity value at one-minute intervals for a total of three readings. The viscosity measurements are in units of centipoises (cP). Convert cP to Pascal·seconds (Pa·s) by dividing cP by 1000.

16. The following items should be reported:

- test temperature,
- spindle number,
- spindle speed, rpm, and
- viscosity, Pa·s, nearest 0.1 Pa·s.

17. Total time, including sample and equipment preparation, to perform this procedure should be less than an hour for a skilled operator.

18. To clean the sample chamber, remove it from the thermo-container using the extraction tool and discard the sample. Allow the hot sample chamber to cool about 5 minutes. Clean the sample chamber using mineral spirits or other suitable solvent. The sample chamber should be wiped with a clean cloth so that no solvent residue remains. Acetone is effective in removing solvent residue.

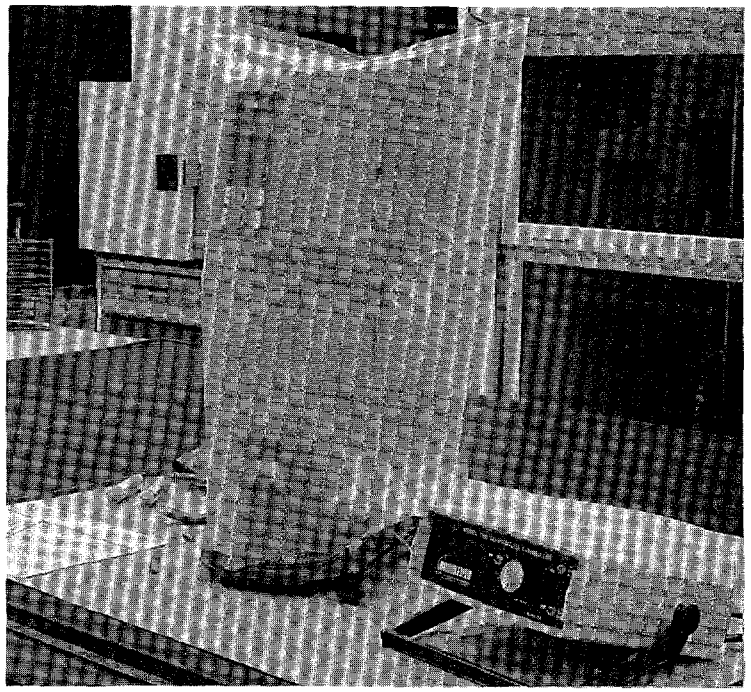


Figure 11. Rotational Viscometer

National Asphalt Training Center

TECHNOLOGY APPLICATIONS

Rolling Thin Film Oven

Test Method for Aging Asphalt Binders Using a Rolling Thin Film Oven

This document outlines the basic steps in aging asphalt binders using a rolling thin film oven (RTFO) according to AASHTO T 240. It provides an illustrated overview, but it is not intended to replace the standard test method which contains detailed information pertaining to all aspects of the test.

1. The rolling thin film oven (RTFO) aging procedure is a conditioning step that simulates construction aging of asphalt binders. Additional tests are performed on the residue from the test. The rolling thin film oven (Figure 1) consists of an oven chamber with a vertical circular carriage. Sample bottles rest in the carriage and the assembly rotates about the carriage center. A fan circulates air in the chamber. At the bottom of the rotation, an air jet blows hot air into the sample bottle.

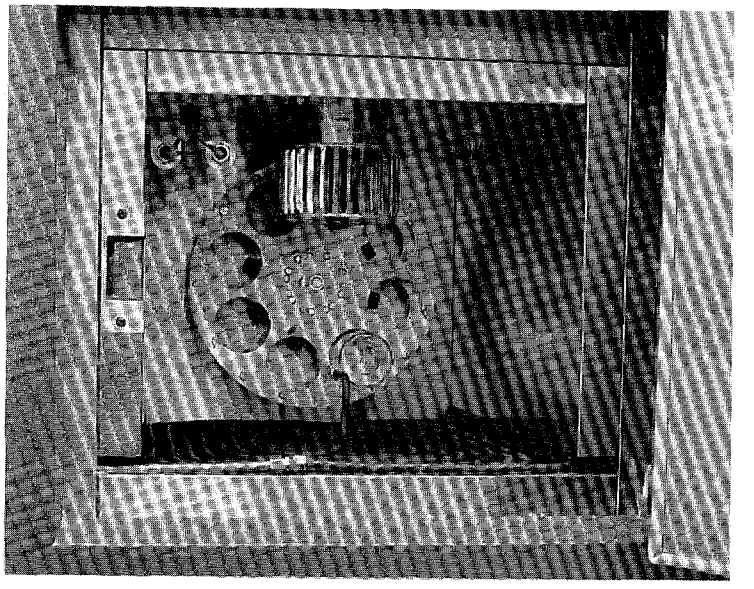


Figure 1. Inside of Rolling Thin Film Oven

2. Turn on the oven and all associated devices (e.g., carriage, fan, etc.) at least 16 hours prior to testing.

3. Heat at least 350 g of asphalt binder sample in a suitable container until the sample is sufficiently fluid to pour. Occasionally stir the sample to ensure homogeneity.

4. While sample is heating, weigh two bottles on a scale accurate to the nearest 0.001 g. These will be used for mass change determination (Figure 2).

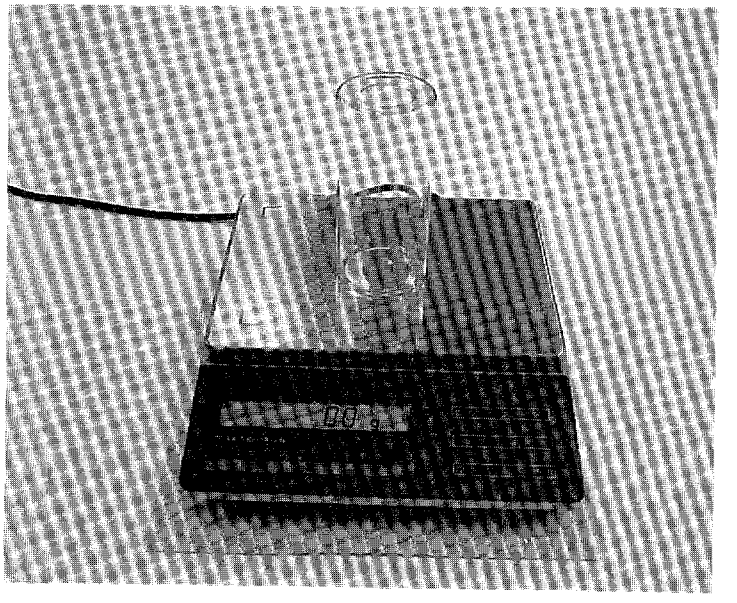


Figure 2. Bottle Prior to Loading Specimen

5. Pour into each bottle 35 ±0.5g of binder (Figure 3). Cool to room temperature.

6. Weigh to the nearest 0.001 g the mass loss samples and bottles.

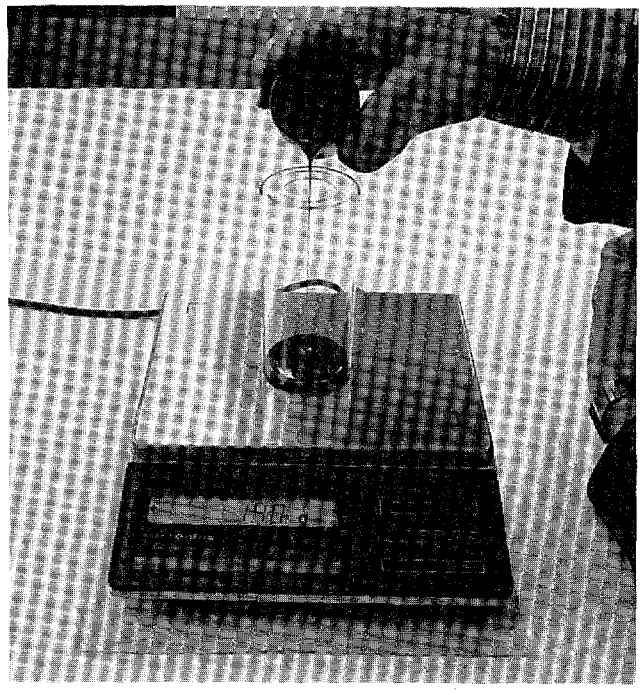


Figure 3. Weighing Specimen Into Bottle

7. With the oven at operating temperature, 163° ±0.5° C, place bottles containing asphalt into the circular carriage Figure 4). Fill any unused spaces with an empty bottle. Oven temperature is displayed by a thermometer suspended in the oven and will drop during placement of the bottles.

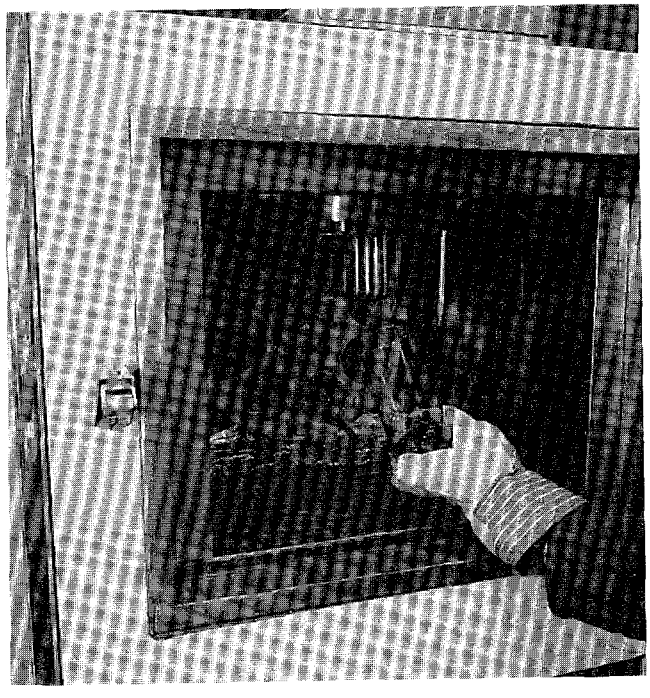


Figure 4. Placing Bottle in Oven

8. Close the door and begin rotation of carriage assembly at a rate of 15 ±0.2 rpm. Start the air flow at a rate of 4000 ± 200 ml/min.

9. Maintain the samples in the oven with the air flowing and carriage rotating for 85 minutes. The proper test temperature must be regained within the first 10 minutes.

10. After the 85-minute conditioning period, turn the oven off and stop the carriage, air flow, and circulating fan.

11. Remove the bottles, one-by-one, keeping them horizontal as they are removed from the carriage to prevent spillage and to facilitate immediate pouring (Figure 5). After each bottle is removed, pour the sample from the bottle into a suitable container. The container should have a large enough capacity to hold all material to be tested. Scrape residue from the bottle to recover material to be tested (see note after step 15). Bottles should be thoroughly coated. Figure 6 shows the three stages of the bottles during the test: empty, with loaded specimen, and with conditioned specimen.

12. For mass change determination, set the designated bottles aside and allow to cool to room temperature.

13. Weigh the mass change bottles containing sample to the nearest 0.001 g and record the weight. Compute the mass change using the equation shown. Discard and do not test the residue from these two bottles.

$$\% \text{ change} = \frac{\text{(orig mass-final mass)}}{\text{orig mass}} \times 100\%$$

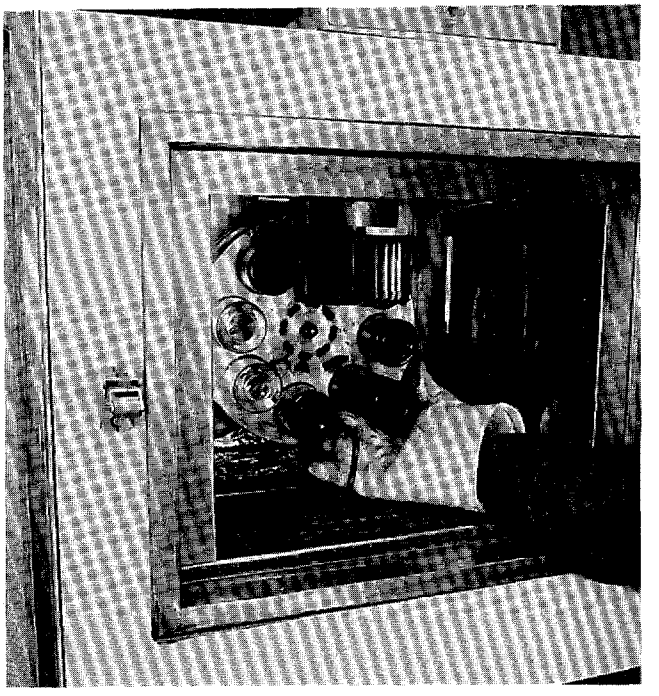


Figure 5. Removing Specimens from Oven

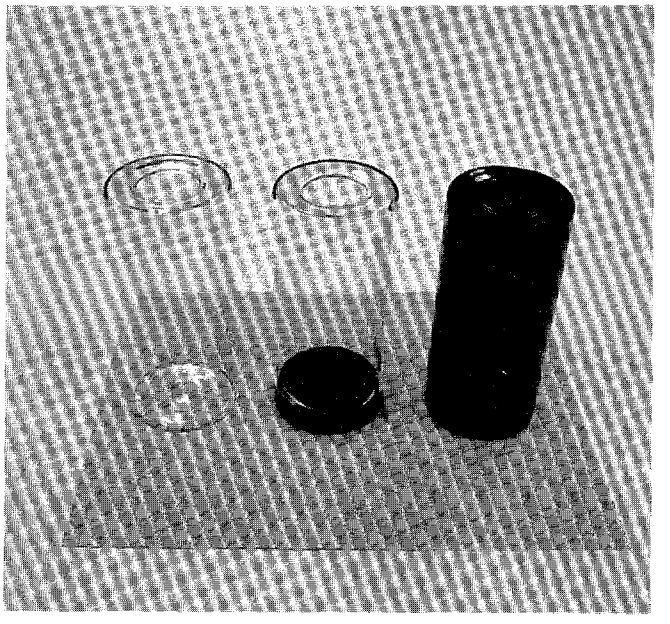


Figure 6. Bottles at Various Stages During Test Procedure

14. The only test result reported is average mass change from the two bottles used for this purpose. All specimens from the same sample are combined, after the RTFO procedure, into a single sample from which specimens can be secured for further testing.

15. Clean the bottles by filling them with mineral spirits or other suitable solvent and allowing them to soak. Decant the solvent and rinse thoroughly with clean solvent. Remove any solvent residue by placing a small amount of acetone in the bottle and wiping with a clean cloth.

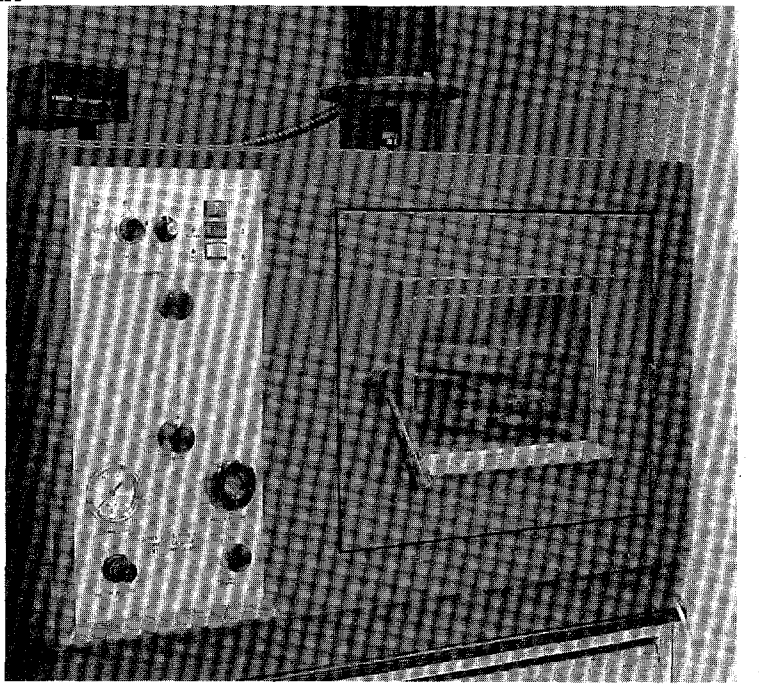
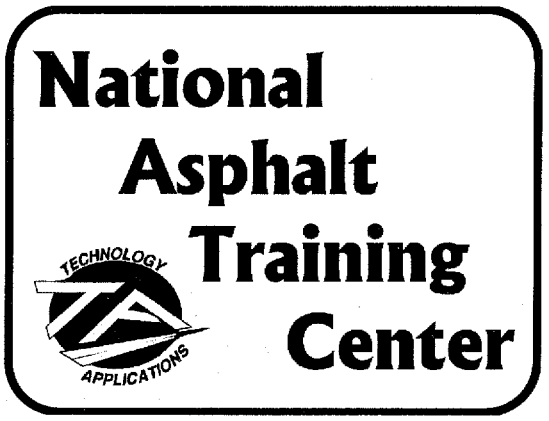


Figure 7. Rolling Thin Film Oven

(Note: The current AASHTO protocol does not allow scraping RTFO bottles to recover binder. However, it may be necessary to scrape the residue to recover sufficient binder for further testing.)



Pressure Aging Vessel

Test Method for Aging Asphalt Binders Using a Pressure Aging Vessel

This document outlines the basic steps in aging asphalt binders using a pressure aging vessel (PAV) according to AASHTO Provisional Method PP1. It provides an illustrated overview, but it is not intended to replace the standard test method which contains detailed information pertaining to all aspects of the test.

1. The pressure aging procedure is a conditioning step that simulates in-service aging of asphalt binders. Additional tests are performed on the residue from the test. The pressure vessel and its components are shown in Figure 1. The pressure vessel oven and pressurized air supply is shown in Figure 2.

2. All pressure aging is accomplished on material that has been previously aged in a rolling thin film oven. Following oven aging, the binder sample should be combined in a single container.

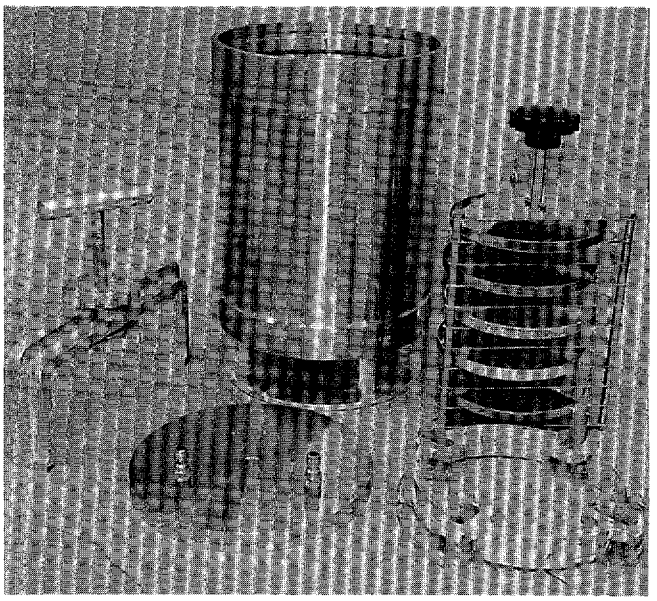


Figure 1. Pressure Vessel Components

3. Place the pressure vessel in the PAV oven that is preheated to a temperature approximately 10° to 15° C higher than the conditioning temperature to be used. Note that there are two temperature displays in the PAV procedure: oven temperature and internal vessel temperature. The oven temperature is displayed by means of a digital indicator, usually on the front of the oven. The internal vessel temperature is measured with a probe that is mounted through the lid of the pressure vessel. This temperature is displayed resolute to 0.1° C on a digital indicator, a continuous chart recorder, or computer interface that monitors vessel temperature during the 20-hour conditioning phase.

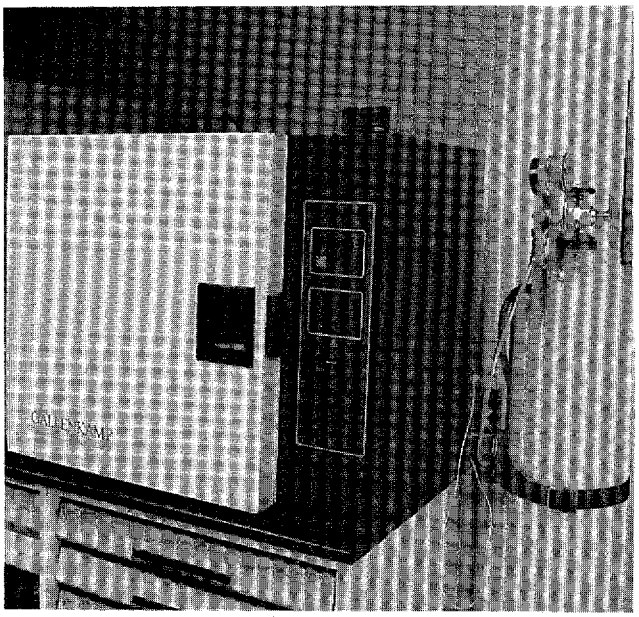


Figure 2. Oven and Pressurized Air Supply

4. Place the container and sample in an oven at approximately 135° C. The sample should be heated to achieve a viscosity of 0.5 Pa·sec, which is about the viscosity of motor oil. While heating, the sample should be periodically stirred to remove any entrapped air and ensure homogeneity.

5. Weigh 50 ± 0.1 g of sample into a PAV pan to to yield a film thickness of 3.2 mm ± 0.1 mm. Repeat this procedure until enough pans are filled to age sufficient material for subsequent testing (Figure 3).

6. Place the pans containing binder samples in the sample rack. A sample rack is shown in Figure 4. The handle inserts and locks into the top of the sample rack to facilitate handling of the rack

7. The next steps involve loading samples into the pressure vessel and preparing the assembly for testing. These steps should be accomplished as quickly as possible so that the vessel and oven lose as little heat as possible.

8. Remove the hot pressure vessel from the oven and quickly place the sample rack in the vessel.

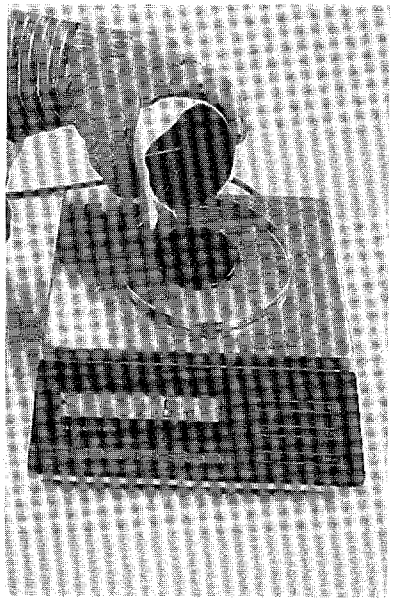


Figure 3. Weighing Sample in Pan

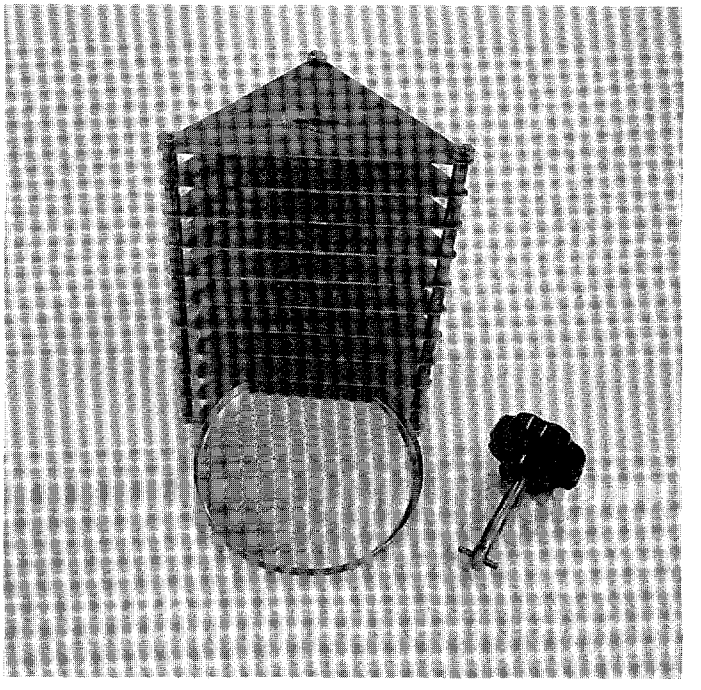


Figure 4. Sample Rack with Handle

9. Lower the pressure vessel lid into place. This can be accomplished manually or by using the t-handle which helps in securing and removing the lid. Figure 5 shows the vessel lid with the t-handle affixed. In this figure, the lid is resting on an empty paint can. This protects the temperature probe that is protruding from the bottom of the lid when the lid is not in use. The threaded rod on the t-handle is screwed into the hole in the vessel lid. The star nuts shown serve to raise and lower the lid. Figure 6 shows the vessel lid and t-handle in position on top of the vessel. When placing the lid on top of the vessel, be careful no to damage the temperature probe. In Figure 6, the vessel lid has not yet been lowered. Lower the lid by turning the upper star nut counter clockwise. Figure 7 shows the vessel lid in place without the t-handle

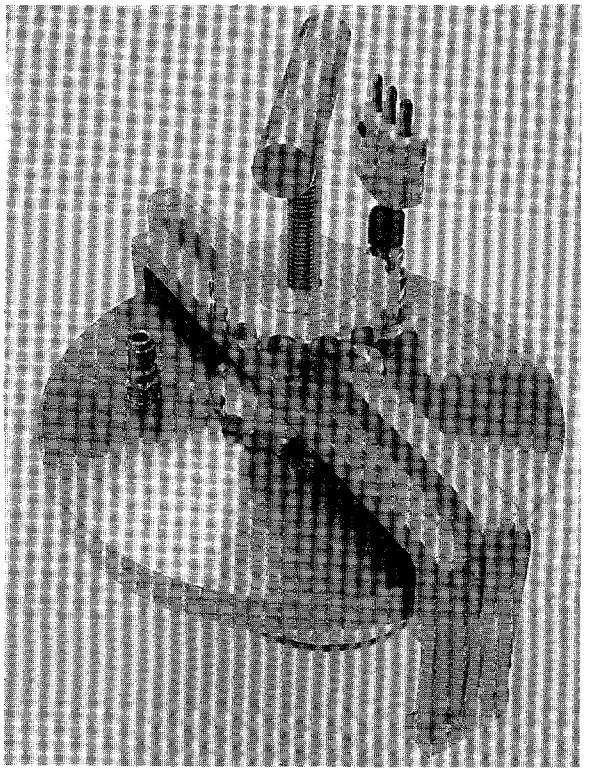


Figure 5. Pressure Vessel Lid with T-Handle Attached

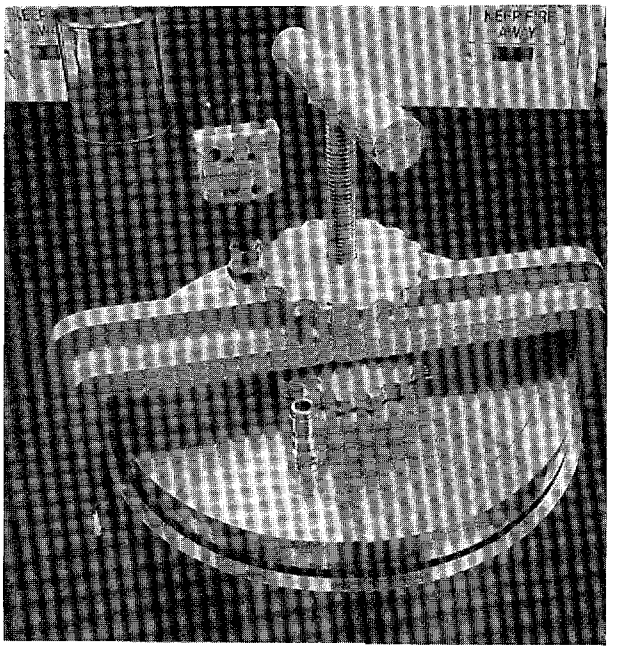


Figure 6. Vessel Lid with T-Handle on Top of Vessel

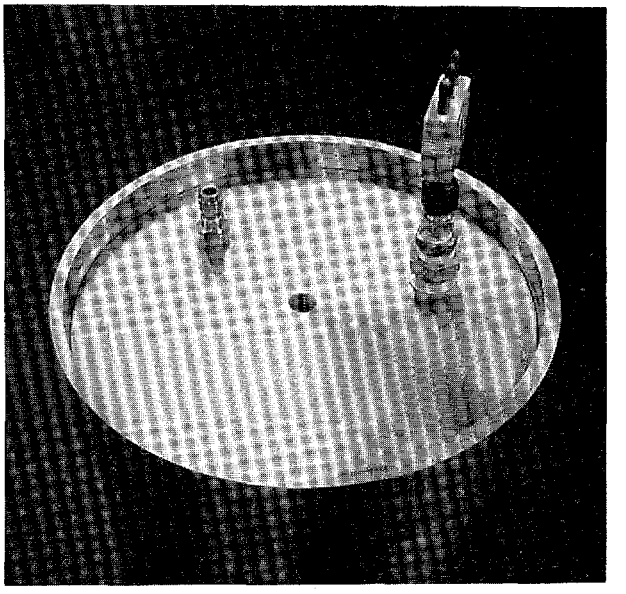


Figure 7. Vessel Lid in Place

10. Loosely place the shear ring assembly (Figure 8) on top of the lid. Insert the bronze shear ring segments into the key way that is machined into the inner wall of the vessel. This is accomplished by aligning and pushing them outward so that the bronze segments easily fit into the key way.

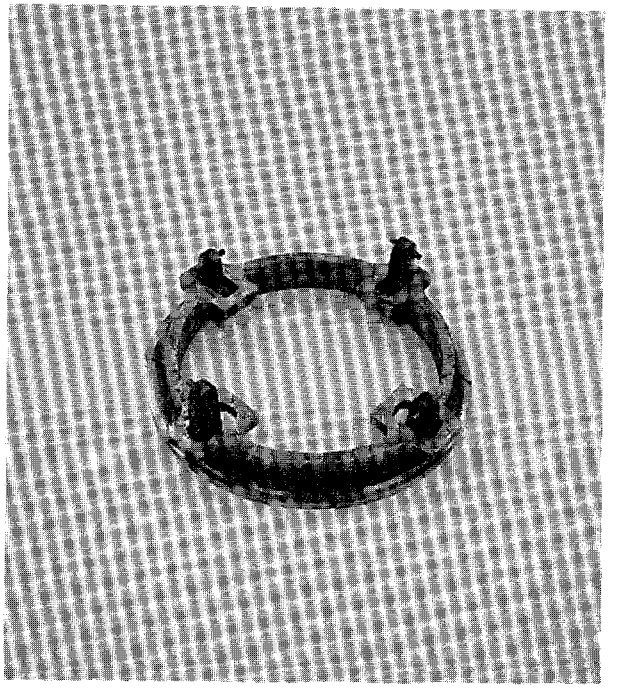


Figure 8. Shear Ring Assembly

11. Push down on the aluminum lock ring so that it is properly positioned to secure the shear ring segments in the key way. Figure 9 shows a schematic of how the shear rings secure the vessel lid. Figure 10 shows the top of the pressure vessel with the lid held securely in place with the shear ring assembly.

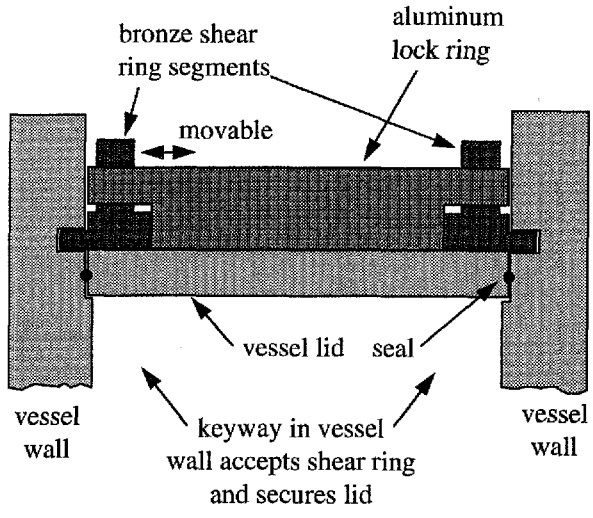


Figure 9. Vessel Lid Secured with Shear Rings

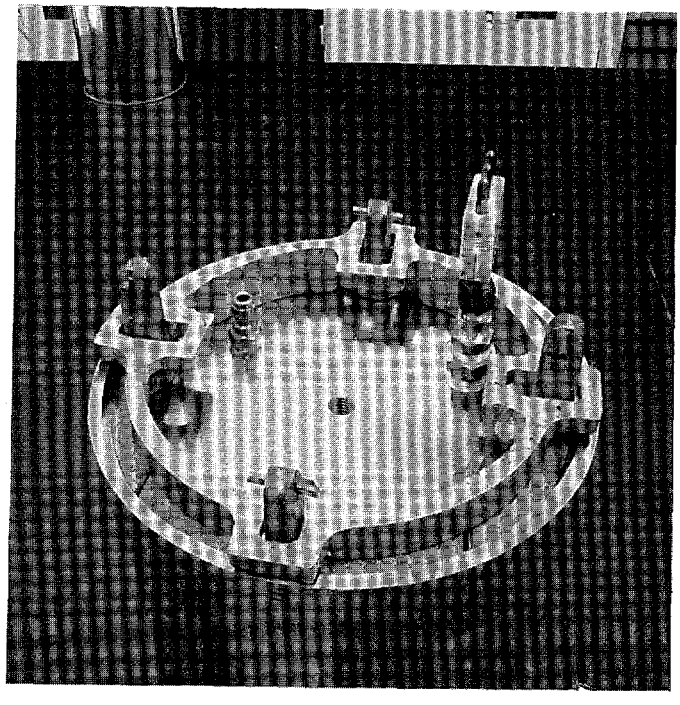


Figure 10. Shear Ring Assembly in Place

12. Place the vessel in the hot oven. Initially position the vessel so that the pressure hose and temperature probe leads can be attached. Figure 11 shows the vessel as it is being placed in the oven.

13. Attach the pressure hose using the quick release coupling. Attach the temperature probe leads as necessary. The pressure hose and temperature probe have been coupled.

14. Reposition the vessel on top of the base support so that it is well supported and level in the center of the oven. Figure 12 shows the vessel in the oven, ready for conditioning to begin.

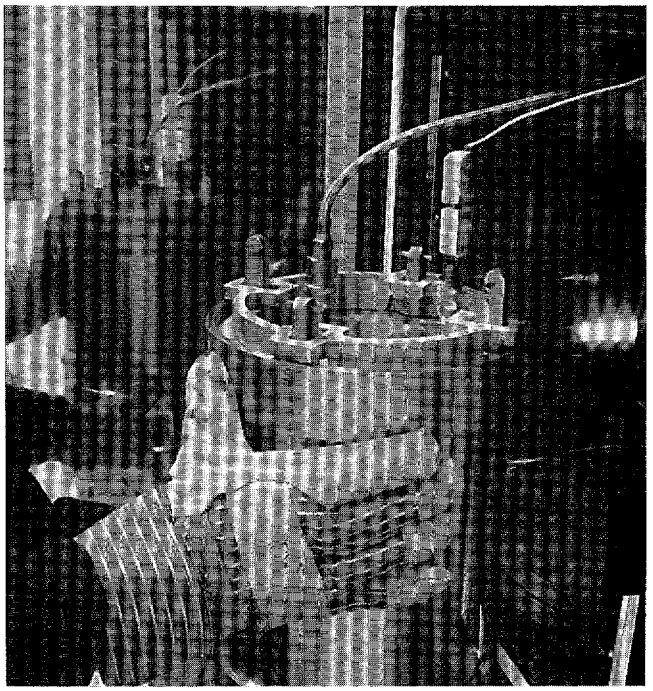


Figure 11. Hot Pressure Vessel Going Into Oven

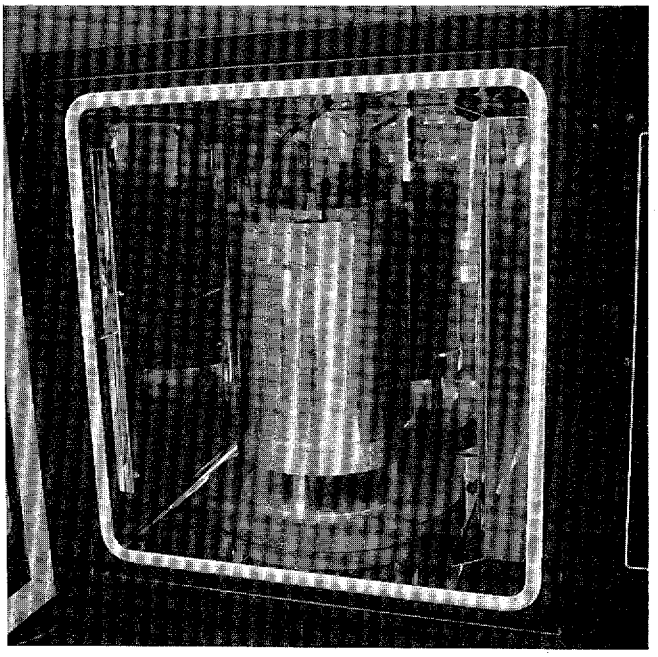


Figure 12. Pressure Vessel in Oven

15. Close the oven door and set the oven at the desired conditioning temperature.

16. Monitor the vessel temperature. When the internal pressure vessel temperature rises to within 2° C of the desired temperature, slowly apply pressure by opening the regulator valve and check for leaks. When pressure is first applied start timing the conditioning process and start temperature recordation. The regulator and pressure indicator are shown in Figure 13. Gradually increase the pressure until 2070 kPa is indicated on the analog scale on the regulator gage. This gradual application of pressure should take approximately 1 to 3 minutes.

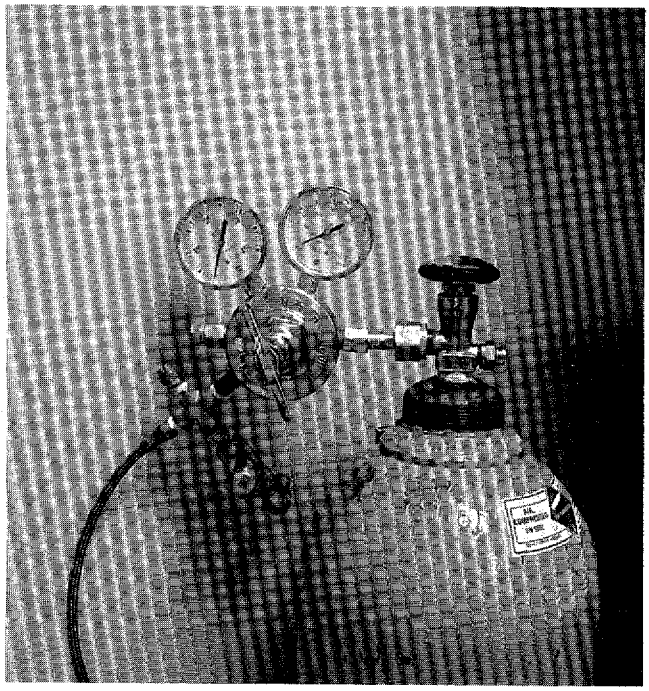


Figure 13. Pressure Regulator and Analog Pressure Gage

17. Allow the binder samples to condition for 20 hours $\pm$ 10 minutes.

18. The following day, observe the continuous record of vessel temperature. If the temperature fluctuated more than 0.5° C above or below the target test temperature for more than a 10 minute interval, the conditioning is considered invalid and the samples should be discarded.

19. At the end of the conditioning period, turn off the oven, digital vessel temperature display, and chart recorder.

20. Close the valve on top of the compressed air bottle. At this point, the vessel remains pressurized. Using the slow pressure release valve, reduce the vessel pressure to equalize intermal and external vessel pressure. The slow release valve is shown in Figure 14 and is the cross shaped device immediately downstream from the regulator. This step should be performed in 9 ± 1 minutes. Do not include this release period as part of the conditioning period.

21. Open the oven and uncouple the pressure hose and vessel temperature leads. Remove the vessel from the oven.

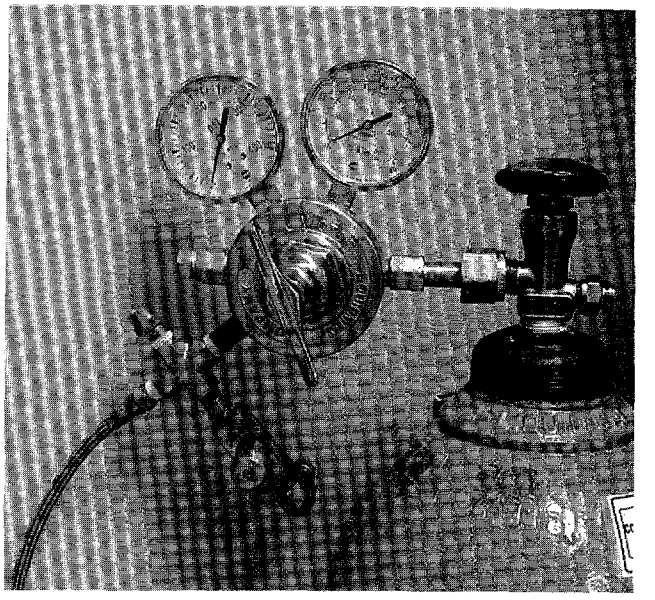


Figure 14. Slow Pressure Release Valve (cross-shaped device to the left of air regulator)

22. Dismantle the shear ring assembly and remove the vessel lid using the t-handle. Remove the sample rack from the pressure vessel.

23. Combine the residue from the pans into a single container (Figure 15). Pour as much residue as possible from each pan into the container and use a flat tool such as a putty knife to remove the any remaining material from the pan. Note that pressure aging often incorporates significant quantities of air into the binder samples. Prior to the transfer of binder samples from the pans to the container, cover the pan and place them in an oven at 163° C for 30 minutes with occasional stirring to allow air bubbles to escape.

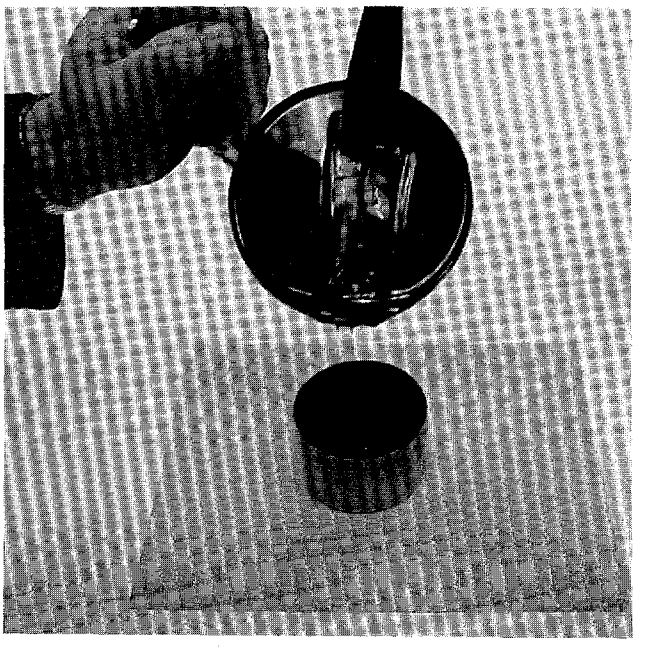


Figure 15. PAV Residue Transferred to Single Container for Later Testing

24. Store the container at ambient temperatures until ready for use. If not immediately used, a lid should be placed on the container to protect the sample from accidental contamination.

25. For each test, the following information needs to be reported:

- sample identification,
- aging test temperature, nearest 0.1° C,
- maximum and minimum aging temperature recorded, nearest 0.1° C,
- total time during aging that temperature was outside the specified range, nearest 0.1 minute, and
- total aging time, hours and minutes.

26. Note that if the temperature shown by the continuous chart recorder fluctuates by more than 0.5° C from the target aging temperature for more than 10 minutes, the test is invalid and the material should be discarded.

27. Clean the PAV pans by soaking them in mineral spirits or other suitable solvent. In all cases, the pans should be wiped with a clean cloth and so that no solvent residue remains. Acetone is effective at removing solvent residue.

28. The pressure vessel and sample rack periodically should be cleaned by wiping with a clean cloth, slightly moistened with solvent. To eliminate any possibility of sample contamination, remove any solvent residue with acetone.

29. The oven or temperature chamber should be periodically checked for levelness. A suitable shelf, stand, or other device should be used in the bottom of the oven to maintain levelness of the pressure vessel. The shelf should be strong enough to resist bending when supporting the weight of the vessel. It also protects the oven floor from being damaged by repeated loading and unloading of the vessel.

30. Equipment calibration and verification should be performed periodically. This includes calibration of the resistance thermal detector (RTD) used to monitor internal vessel temperature, the pressure gauge, and pressure regulator. Calibration procedures are provided in the test method.

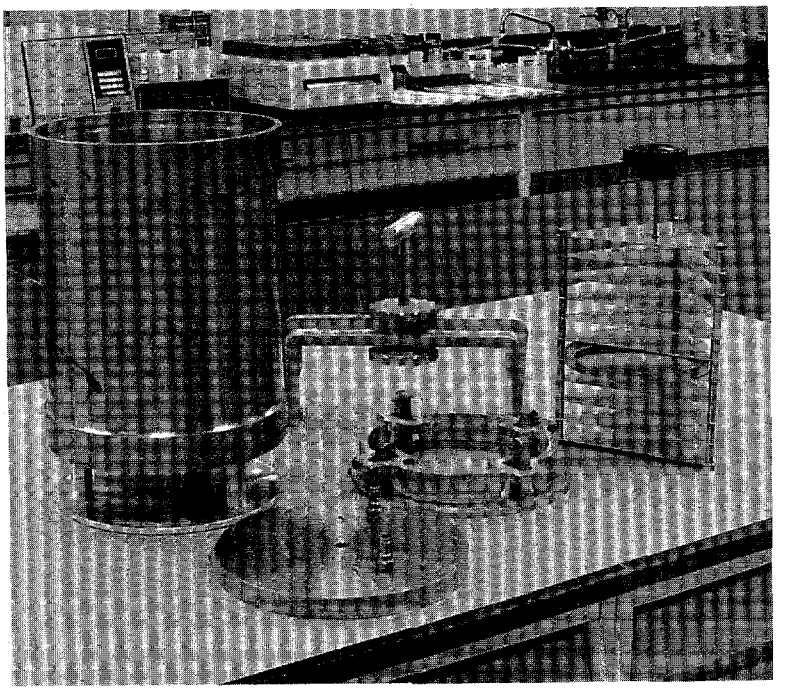
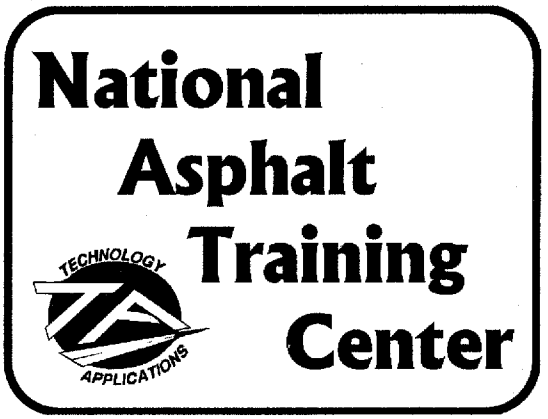


Figure 16. Pressure Aging Vessel and Components



Bending Beam Rheometer

Test Method for Measuring Creep Stiffness (S) and Slope (m) of the Log Stiffness versus Log Time Curve of Asphalt Binders at Low Temperatures Using the Bending Beam Apparatus

This document outlines the basic steps in measuring the creep stiffness (S) and the logarithmic creep rate (m-value) of asphalt binders according to AASHTO Provisional Method TP1. It provides an illustrated overview but is not intended to replace the standard test method which contains detailed information pertaining to all aspects of the test.

1. This procedure outlines determination of flexural creep stiffness of asphalt binders. The apparatus consists of a bending beam rheometer (BBR), a controlled temperature liquid bath, and a computer that is used to control the rheometer and acquire data. The test is performed on binders that have been aged in a pressure aging vessel. A typical bending beam apparatus is shown in Figure 1.

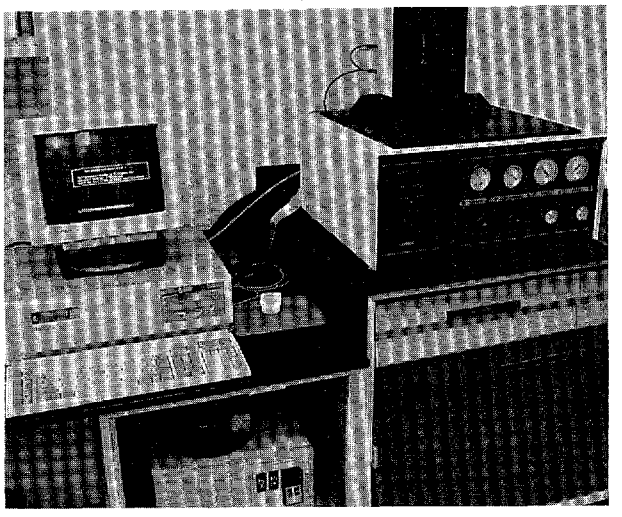


Figure 1. Bending Beam Apparatus

2. Turn on the rheometer air system by opening the supply regulator. The regulator is a valve affixed to the central laboratory air system and is normally located close to the rheometer. In many cases, the valve is part of a combination regulator/water filter system and is not part of the rheometer itself.

3. Turn on cooling system (Figure 2) and set desired test temperature. Refer to the operator's manual to determine the proper way of adjusting temperature.

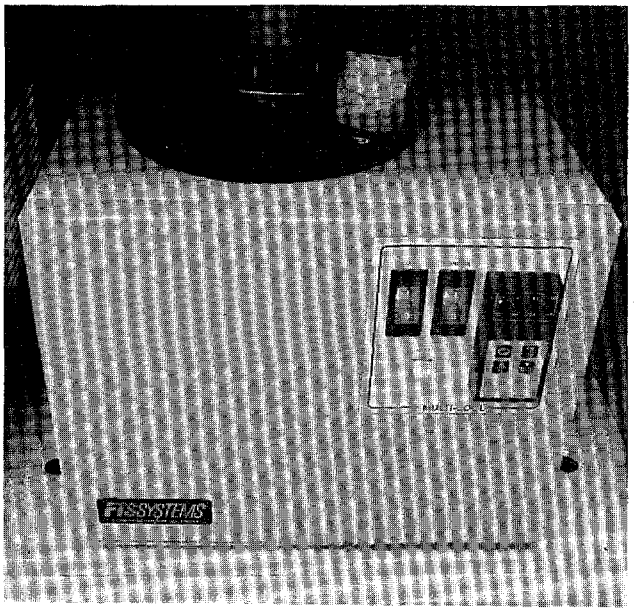


Figure 2. Refrigeration Unit with Front Controls

4. Note: For very low test temperatures (approximately less than -12° C), it may be desirable to turn on the cooling unit at the end of the day before the test is to be performed since it takes several hours for the bath fluid to achieve temperatures this low. It will be necessary, in some cases, to leave the unit running unattended for long periods.

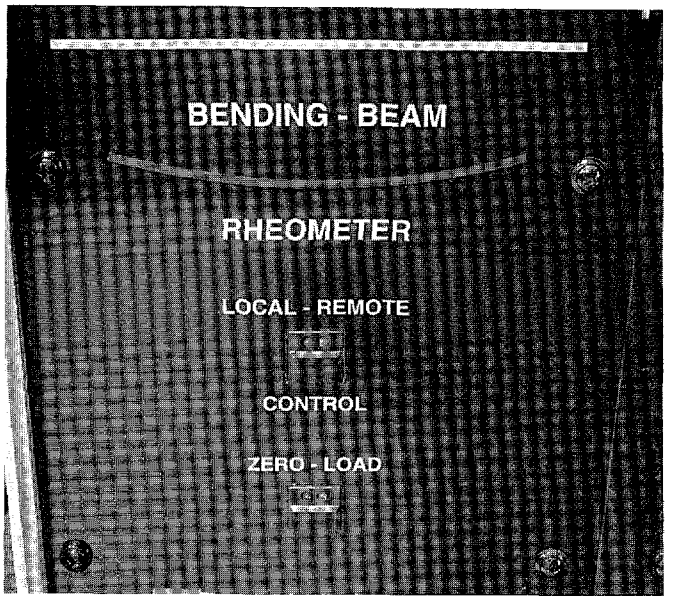


Figure 3. Local/Remote and Zero/Load Switches

5. Turn on personal computer and run program that controls test. At the conclusion of this step, the control software main menu should appear. Turn on rheometer. Push the local-remote button on the front of the rheometer (Figure 3) so that the red light indicates the rheometer is in the local mode. Push the zero-load button so that the red light indicates the rheometer is in the zero mode. Figure 4 shows the front of a rheometer. The controls on the left are the local/remote and zero/load. The knobs are air regulators that control zero load and test load. The operator is actuating the zero load regulator.

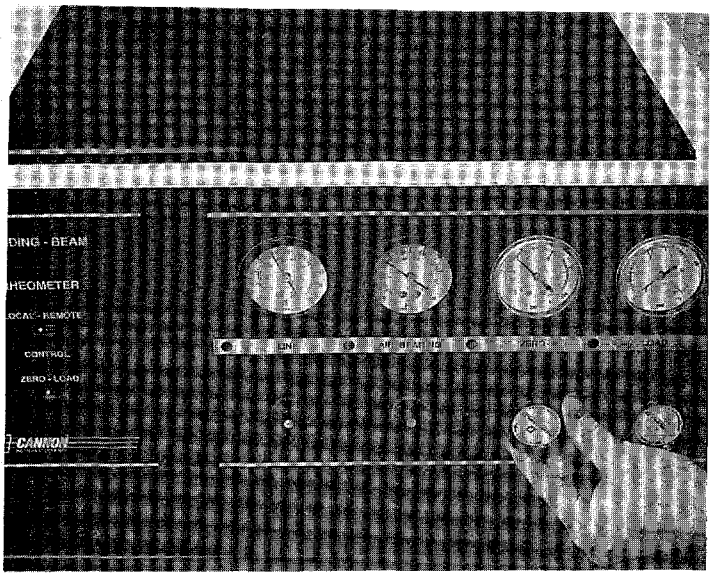


Figure 4. Front of Rheometer Showing Line Pressure and Regulators

6. Place container with binder sample in oven at approximately 135° C. Some residues may require higher oven temperatures, but should never exceed 150° C. The actual temperature used should be that to achieve a viscosity of less than 0.5 Pa·s, which is the approximate viscosity of motor oil. Stir sample occasionally while it is being heated (Figure 5).

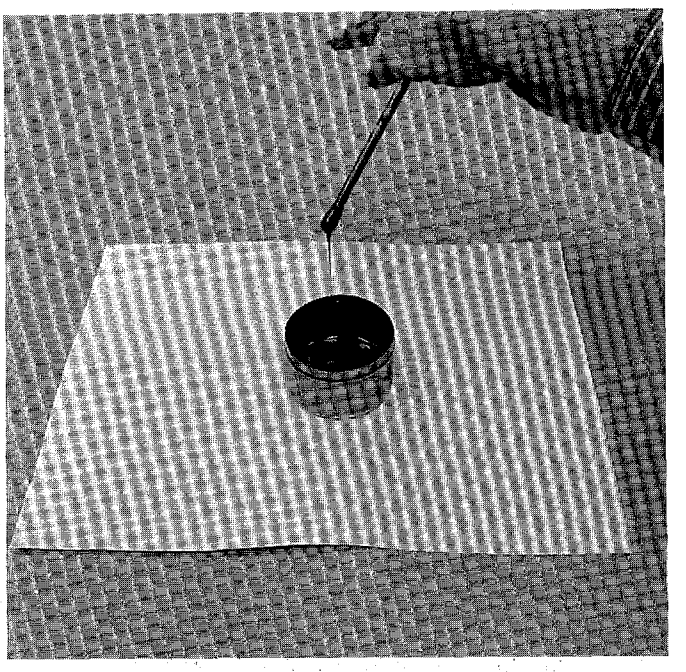


Figure 5. Preparation of Sample

7. While sample is being heated, calibrations of the rheometer should be performed. Items to calibrate include displacement transducer, load cell, and temperature transducer. Rheometer compliance, i.e. the deflection of the rheometer loading frame, is also checked. These activities are controlled by the rheometer software and the operator need only follow the instructions provided on the computer screen. Several calibration items are provided to perform these steps (Figure 6). The circular item on the left is a gage used to calibrate the deflection transducer. The circular weights are 100 grams each and are used in several calibration steps. The thick steel beam is used to check rheometer compliance and the thin steel beam is used for a system confidence check.

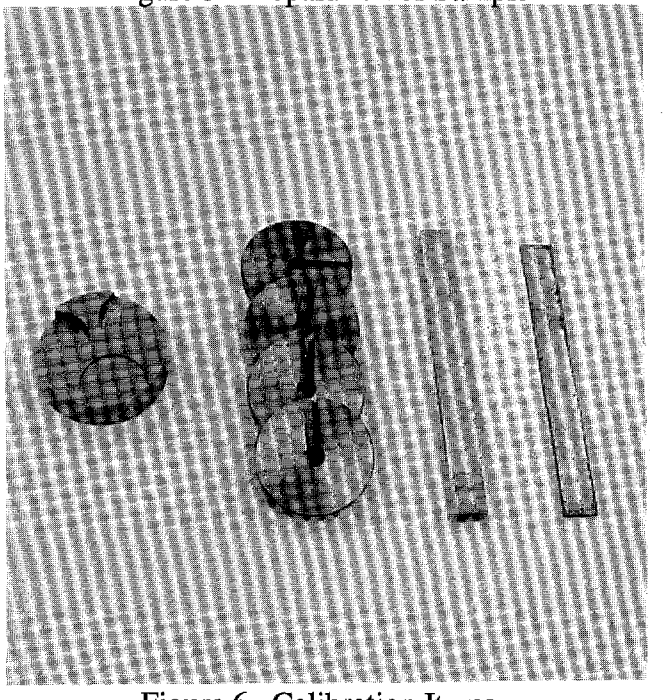


Figure 6. Calibration Items

8. From main menu, select the calibration option. From the calibration menu, select "defl, load, confidence." Calibration directions are provided on the computer screen. A typical procedure is as follows.

9. The first item calibrated is the displacement transducer. This is accomplished by means of a stepped thickness gage of known dimensions and a 100 g weight. The gage is placed over the gage pin that is on the load frame. The gage may be rotated about the pin during calibration. Once the gage is on the pin, lower the platform using the zero regulator (on the front of the rheometer) so that the pin on the bottom of the load platform rests on the uppermost part of the gage. Place a 100 g weight on the load platform. Figure 7 shows gage and 100 g weight in position. A software prompt requires pressing "enter" when this position is achieved.

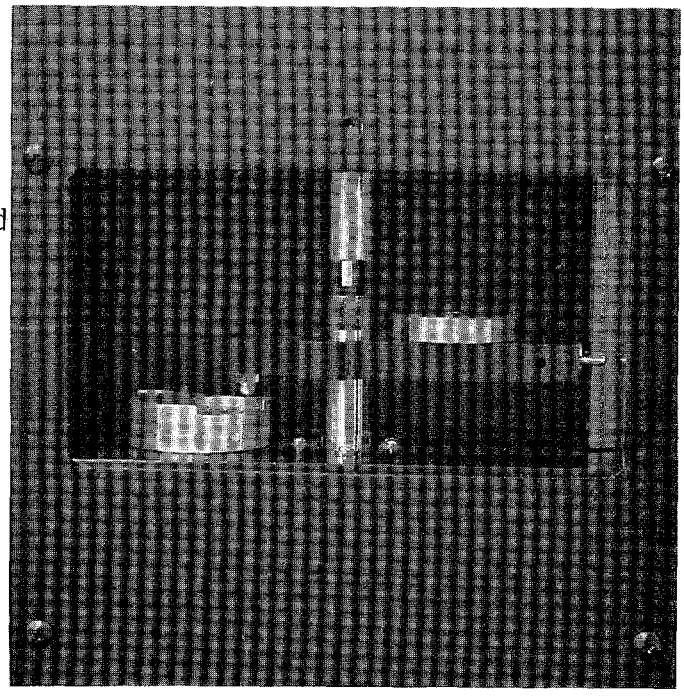


Figure 7. Stepped Gage and 100 Gram Weight in Place During Deflection Transducer Calibration

10. A new computer screen appears with new directions as follows. Using light finger pressure (Figure 8), slightly raise the loading platform, still containing the weight, and rotate the stepped gage so that the lower pin rests in the highest slot. This slot is labeled by a red dot. Allow the pin in the loading platform to rest in this slot. Another software prompt requires pressing "enter" when this step has been accomplished.

11. The previous step is performed two more times using progressively deeper slots, which allows more deflection of the loading platform. At the conclusion of the fourth deflection measuring cycle, the deflection check is complete and system software offers a prompt to proceed to the load cell calibration.

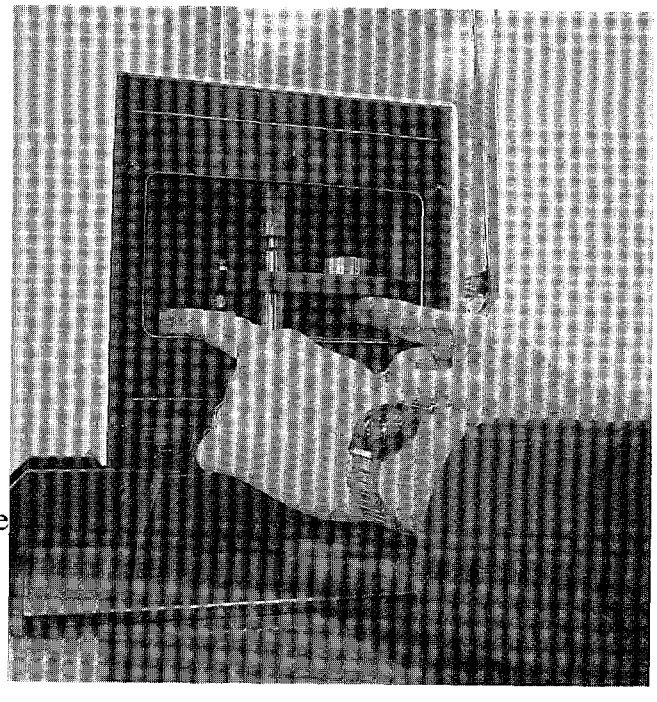


Figure 8. Light Manipulation of Loading Platform

12. The gage block and 100 g weight are removed. Next, center the steel beam (6.350 mm thick) on the beam support. Because the load supports are submerged under the frigid bath fluid grasp and position the beam using forceps. Figure 9 shows the beam in place on the supports with the loading frame out of the bath. When positioning the beam, slightly lift the loading shaft out of the way so it does not interfere with positioning the beam and so the shaft is not damaged. When the beam is in position, lightly rest the shaft on the beam. Adjust the zero load regulator (see Figure 4) so that the shaft is almost floating but still lightly in contact with the beam.

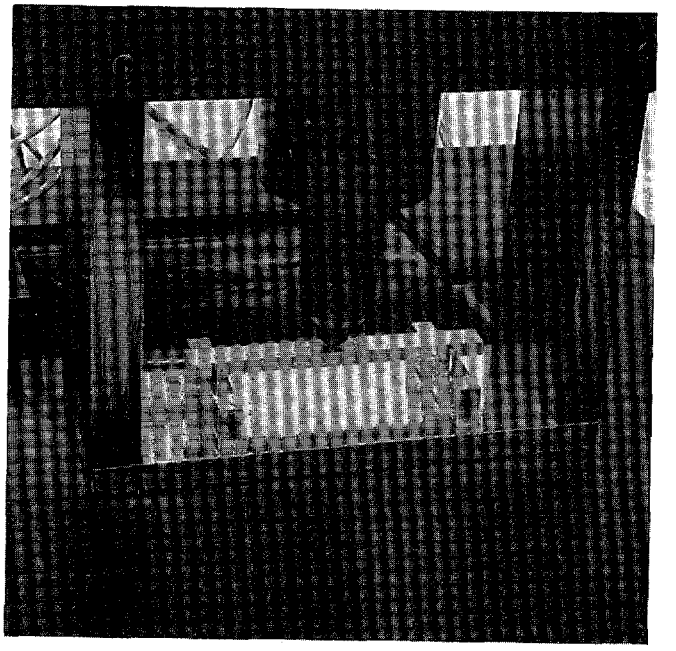


Figure 9. Compliance Check Beam in Position (Loading Frame Out of Water Bath for Clarity)

13. To accomplish this delicate operation, observe the loading platform at the upper end of the shaft. When adjusting the zero load regulator, the shaft will move down and abruptly stop when it contacts the beam. Using the zero load regulator, back the shaft off slightly so that there is no load on the beam yet the shaft is still in contact with the beam. Slight upward finger pressure (Figure 10) on the loading platform will verify this desired condition has been achieved. By slightly raising and lowering the loading platform, the operator should be able to feel the shaft bottom-out on the beam. If rheometer software allows determination of shaft/beam contact, use that approach.

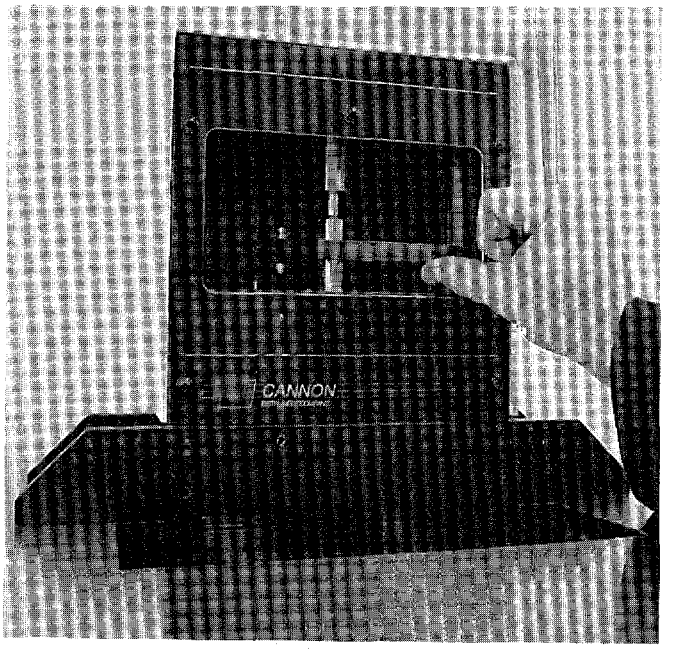


Figure 10. Checking that Shaft is in Contact with Beam by Light Finger Pressure

14. Place a 100 g weight on the loading platform (Figure 11) and allow about five seconds stabilization period to elapse prior to pressing "enter," which tells the computer that the device is prepared to complete the load cell calibration procedure. The software will deliver a screen prompt indicating the need for a second 100 g weight to be placed on top of the first. After the second weight is placed, the operator presses "enter." This cycle of placing weights occurs two more times for a total of 400 g on the loading platform.

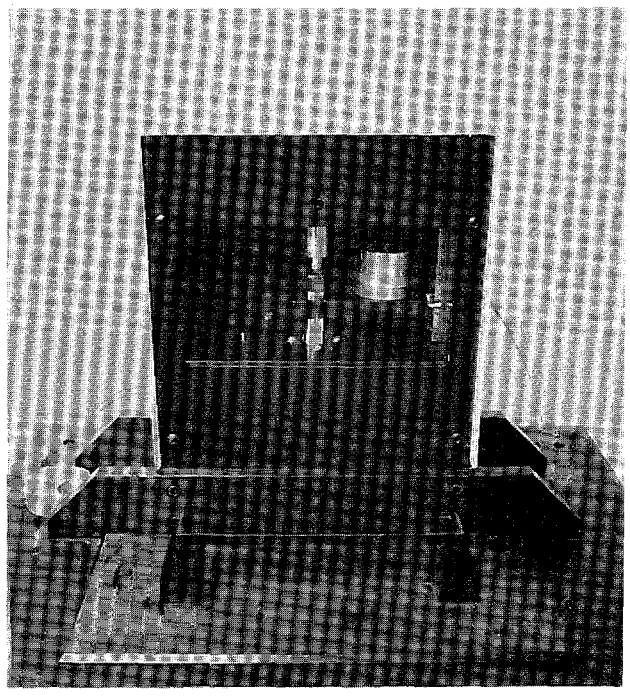


Figure 11. Weights on Load Platform During Compliance Check

15. At the conclusion of this sequence, a computer screen indicates calculated values for deflection constant, load constant, and calculated compliance. These values are compared with those listed on the rheometer calibration certificate or set-up menu. Calibration confirmation continues by pressing "enter."

16. The next step uses a thin steel beam of known stiffness modulus to perform a confidence check of the rheometer system. The local-remote button should be set from local to remote at this time. Remove the four 100 g weights from the loading platform. Also lift shaft and remove the steel beam from the loading supports.

17. Using the same procedure as before, place the 1.3 mm thick (nominal) steel beam on the loading supports. Lower the shaft and place a 100 g weight on the loading platform. The confidence check test is started by pressing "S" on the computer keyboard. In a few seconds, the operator is prompted to place an additional 300 grams (i.e., total of 400 g) on the loading platform followed by "enter." After about 20 seconds, a modulus value for the steel beam is shown and based on this value, the software indicates whether the rheometer has passed or failed the confidence check.

18. If the rheometer fails the confidence check, the operator is prompted to start the calibration procedure over. If the rheometer passes the confidence check, the operator may proceed with testing asphalt beams.

19. Verify the calibration of the temperature detector by placing the bulb of a certified mercury-in-glass thermometer in contact with the resistance thermal detector. Compare the thermometer reading with the computer screen display. If necessary, adjust the displayed temperature to match the thermometer reading. Note that the testing bath should be checked for temperature stability for at least a 65-minute period.

20. Asphalt beams are prepared by using a mold. One type of mold (Figures 12 and 13) consists of aluminum bars, plastic strips, and two release agents, petroleum jelly and a mixture of glycerin and talc. Two of these molds are required in order to make two beam test specimens.

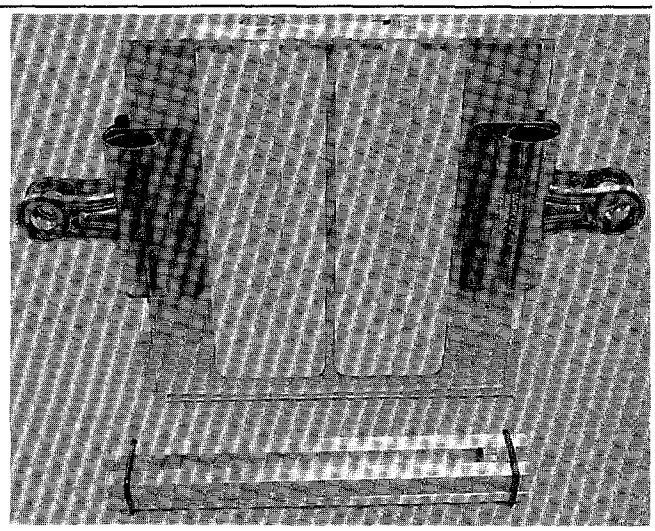


Figure 12. Two Types of Beam Molds

21. The other type of mold (Figures 12 and 14) is composed of silicone rubber and can form two beams. The molds are placed in an aluminum stand with a silicone rubber sheet and piece of glass, which are held in place by clips.

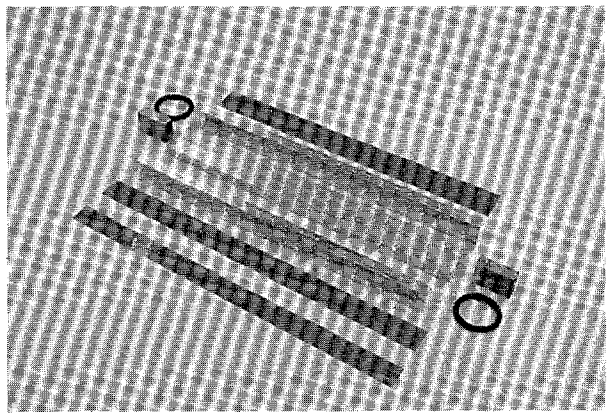


Figure 13. Aluminum Mold Components

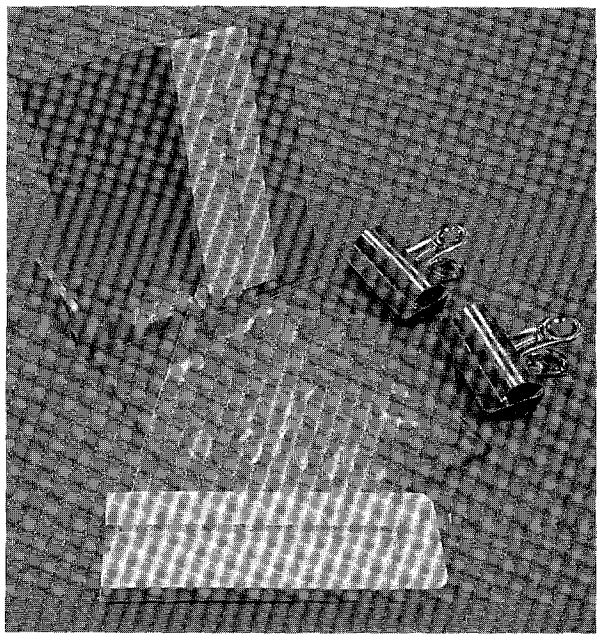


Figure 14. Silicone Rubber Mold Parts

22. Working on a firm level surface, prepare a mold by first applying a thin film of petroleum jelly to the inside of base bar (Figure 15) and inside of two side bars. Place plastic strip onto coated side of bars. The petroleum jelly serves to hold the plastic strip in place.

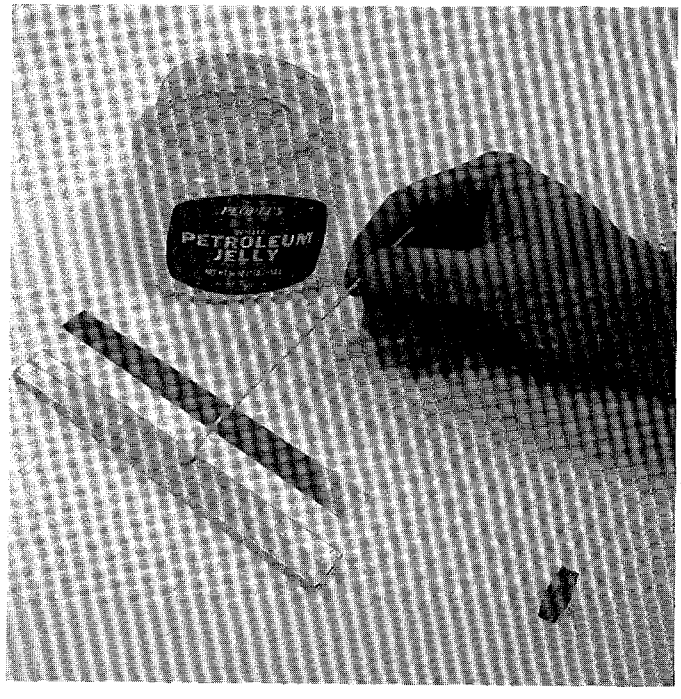


Figure 15. Applying Vaseline to Base Bar

23. To make the glycerin/talc release agent, mix talc and glycerin in a 50 ml beaker or other small container until the mixture has a paste-like consistency.

24. Lightly coat with glycerin/talc mixture, one edge of each of two end pieces (Figure 16).

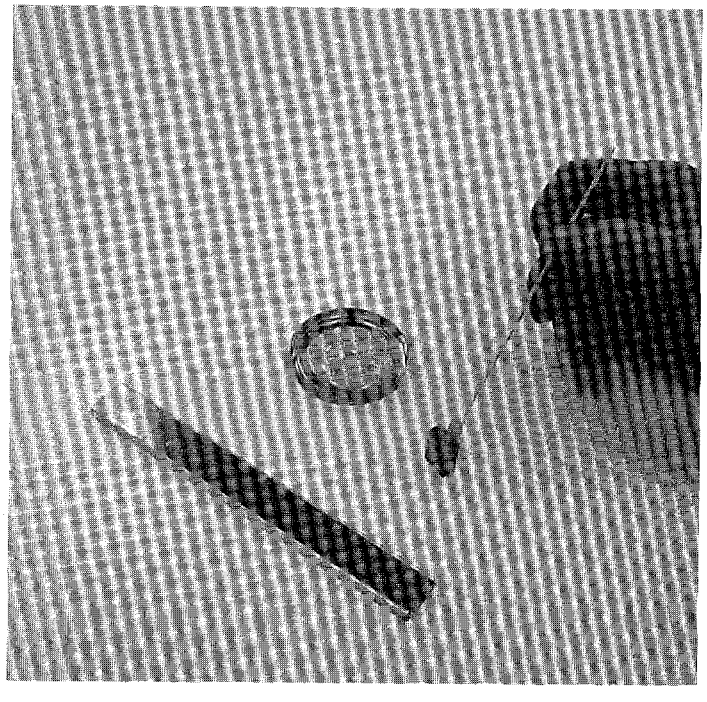


Figure 16. Applying Release Agent to End Pieces

25. Place one side bar onto the base bar. Place two end pieces onto base bar followed by the remaining side bar. Slide assembly to the edge of work surface and slip rubber O-rings over ends to firmly hold assembly together. If plastic strips lose contact with bars, pull on the end of the strips to place them in contact with the bar surface.

26. Prepare another mold as above since two beams are required for a test.

27. Pour sample (Figure 17) in one pass into molds from one side to the other allowing the stream of material to flow ahead and fill mold. When filling, hold sample container 20 to 50 mm above mold. Slightly overfill the mold to allow for later trimming.

28. Allow the mold to cool to room temperature for 45 to 60 minutes.

29. While still in the mold, trim the exposed face of the beam specimen using a hot knife edge such as putty knife (Figure 18). Heat the knife edge for a few seconds over a bunsen burner flame or by placing it on a hot plate. When trimming, use a slow steady motion so that the beam specimen is cut and not pulled so as to not deform or excessively disturb the specimen.

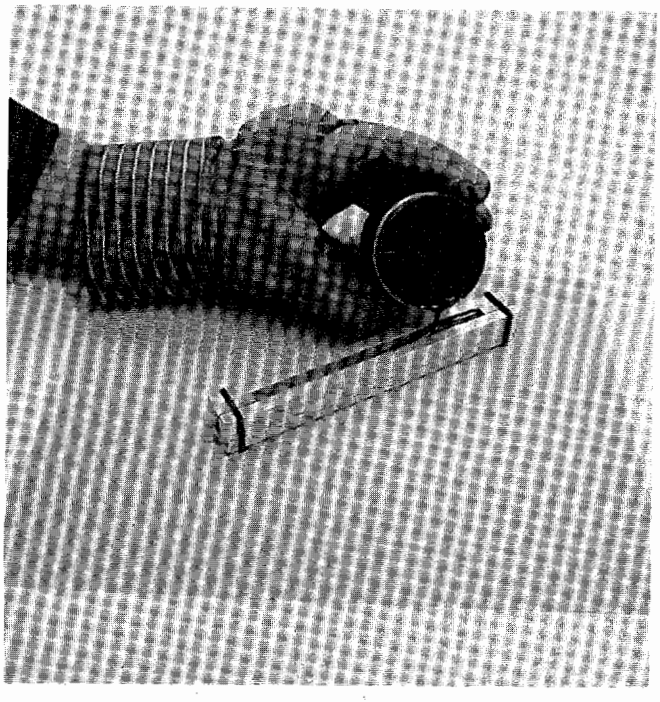


Figure 17. Pouring Sample Into Mold

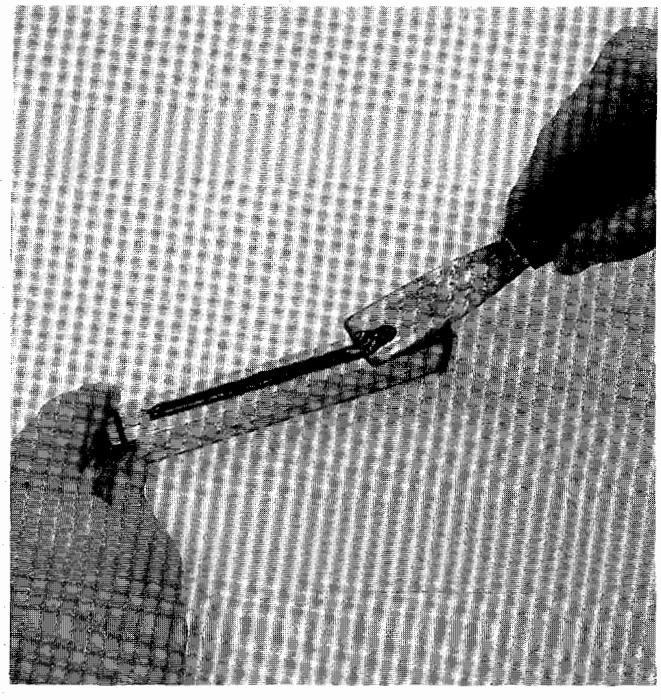


Figure 18. Sample Trimming

30. If necessary, use a sharp implement such as a single-edged razor blade to remove traces of asphalt that may remain on the top surface of the side bars and end pieces (Figure 19). Removing this material will help in the demolding process.

31. While still in mold, place the beam specimens in a freezer for 5 to 10 minutes or an ice bath for 30 to 45 seconds. Do not use the testing bath for this purpose since it may change cause temperature fluctuations. Effective demolding requires that the specimen remain cold. During the demolding process and if necessary, it is reasonable to place the specimen and any remaining mold pieces back into the freezer or ice bath for a few seconds to facilitate ease of removal of mold pieces from the beam specimen.

32. Remove specimens from freezer or ice bath and immediately begin demolding. First, remove O-rings from the end of the assembly. Twist the base bar away and remove it from the assembly. Peel plastic strip away starting at one end and proceeding to the other (Figure 20). Next, gently twist one side bar away (Figure 21) from beam specimen and remove plastic strip as before. Remove the end pieces. Remove the remaining side bar and plastic strip.

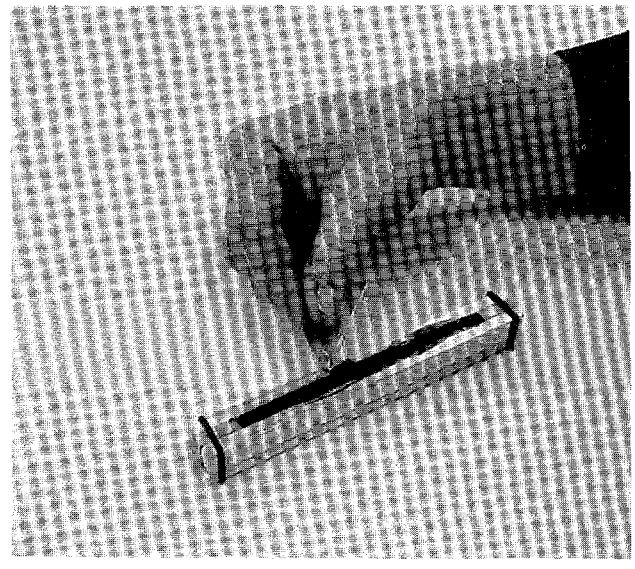


Figure 19. Removing Excess Sample from Top of Side Bar

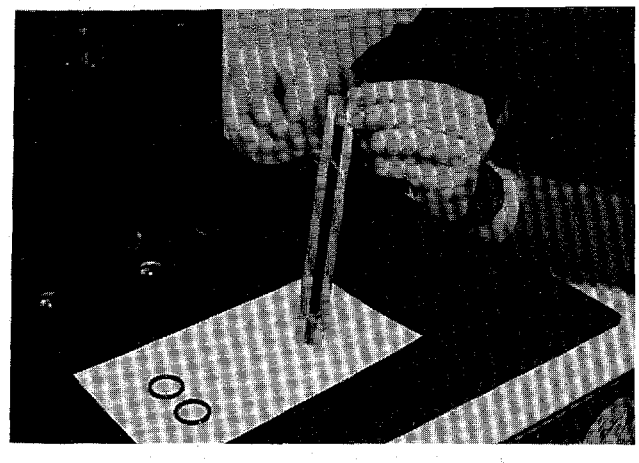


Figure 20. Peeling Bottom Plastic Strip

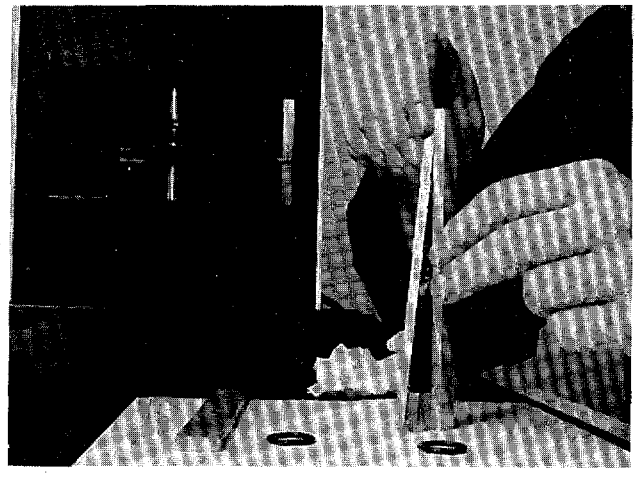


Figure 21. Twisting Side Bar

33. To use the silicone rubber mold, assemble the components as shown in Figure 12. Pour the hot asphalt through the access hole in the top of the molds (Figure 22). Allow the mold to cool to room temperature for at least 45 minutes.

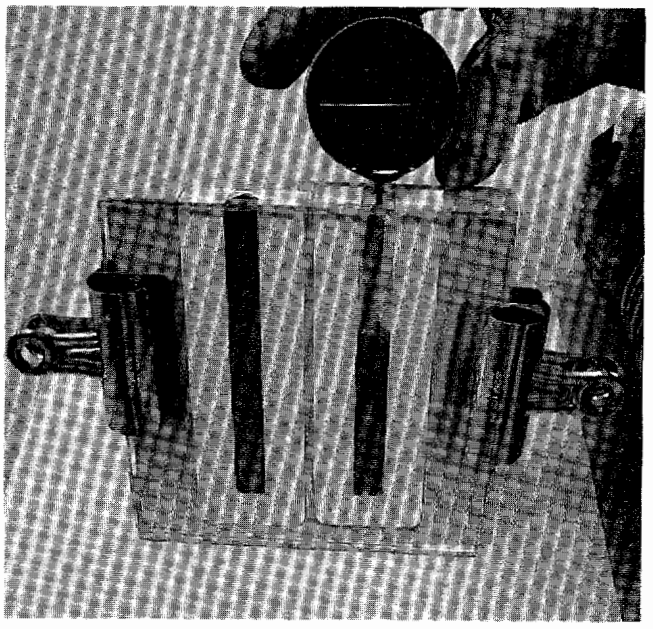


Figure 22. Pouring Sample Into Mold

34. While still in mold, place the beam specimens in a freezer for 5 to 10 minutes or ice bath for 30 to 45 seconds. Do not use the testing bath for this purpose. Remove the clips holding the glass plate and set the plate aside. Peel the silicone rubber sheet away from the beam specimens. Deform the silicone rubber molds so that the beams separate from the molds without deforming (Figure 23).

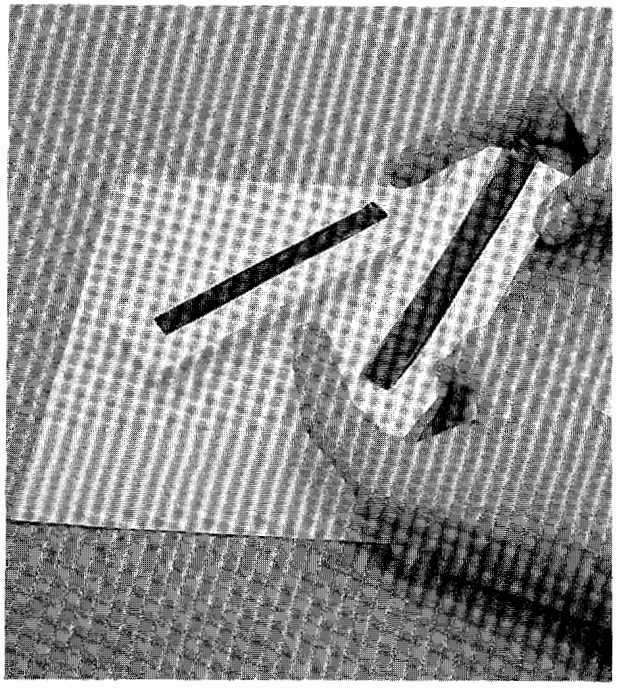


Figure 23. Demolding Specimen from Silicone Mold

35. Submerge the beam specimens in the rheometer bath by placing them under a submerged shelf (Figure 24) or other container so that they do not float to the surface of the bath. The beams will remain submerged, before testing, for a tightly controlled interval of 60 ± 5 minutes.

36. While the samples are submerged, prepare the rheometer for testing as follows. Using forceps, submerge the large steel beam in the rheometer bath and place it onto the beam supports (Figure 9). Slightly raise the loading shaft using finger pressure to fit the steel beam onto the supports. Gently lower the shaft onto to the steel beam.

37. On the computer screen, select "Inputs" from the main menu. The screen will display load, deflection, and temperature. With the local/remote switch still in the local mode and the zero/load switch in the zero mode, adjust the final shaft vertical position by adjusting the zero load regulator (Figure 4). Place 2.5 to 3.5 grams of load onto the steel beam by slightly turning the regulator and observing the indicated load on the computer screen. Verify by slight finger pressure on the loading platform that the shaft is in contact with the steel beam.

38. Change the zero/load switch to the load mode. A green light will appear next to the switch to verify this change. Using the load regulator and by observing the computer screen, apply a 100 ± 5 grams load to the steel beam.

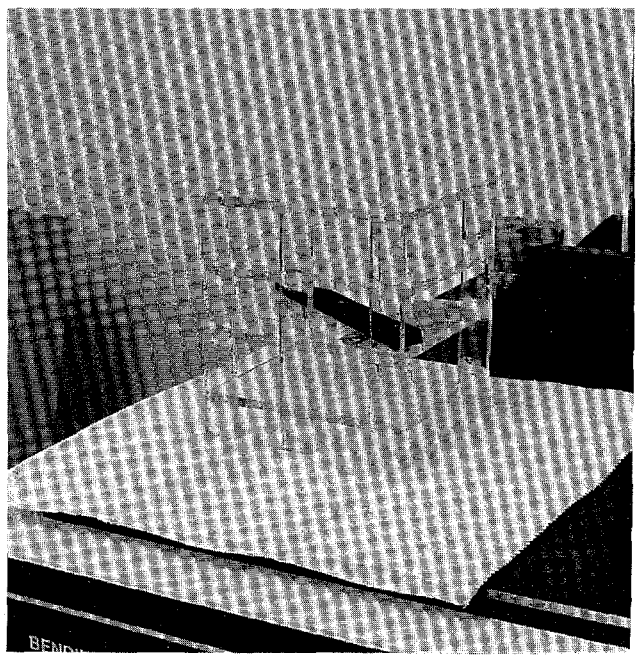


Figure 24. Shelf for Holding Beams (Out of Water Bath for Clarity)

39. Reconfirm the 2.5 to 3.5 gram preload by changing the zero/load switch to the zero mode. Observe the indicated load on the computer screen and verify the 2.5 to 3.5 gram preload. Adjust the preload, if necessary, using the zero load regulator.

40. Reconfirm the 100 gram load by changing the zero/load switch to the load mode. Observe the load on the computer screen and adjust, if necessary, using the load actuator.

41. From the main menu, select "Run Test" and from the computer keyboard input project ID, Operator ID, Specimen ID, and file name. If no file name is entered, the software automatically assigns a file name. From this screen, also input the bath temperature in °C and time beam in bath. (Note that this step does not set the test bath temperature but only allows the temperature to be printed in the final test summary.) The rheometer compliance value shown on the screen is that previously determined from the manufacturer.

42. After test information is input, the beams may be tested as soon as the 60 minute time interval has elapsed. Immediately, prior to testing, it is good practice to verify the preload and test load as described above. A skilled operator can perform the above mentioned load verification procedure in less than 3 minutes.

43. To begin the test, return to the main menu and select the "Run Test" option and verify the test information previously input. From the computer keyboard, hit the esc key and another menu appears. Select "Inputs" from the main menu and on the computer screen to verify that the 2.5 to 3.5 gram preload is evident, which keeps the shaft in contact with the beam. Replace the steel beam with one of the asphalt beam specimens. The asphalt beam should now be in position on the supports. Figure 25 shows an asphalt beam in place on the supports with the loading frame out of the water bath. Gently lower the loading shaft until it is in contact with the asphalt beam.

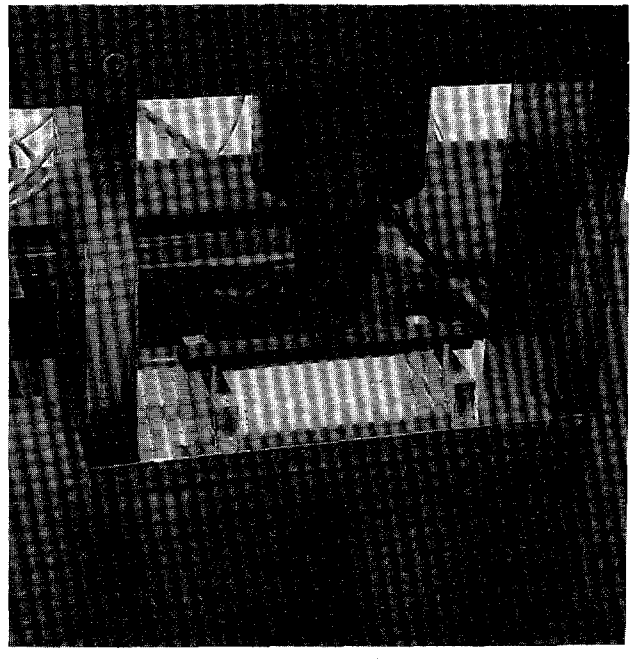


Figure 25. Asphalt Beam in Position (Loading Frame Out of Water Bath for Clarity)

44. Change the local/remote switch to remote mode. A green light will appear indicating the rheometer is in the remote mode.

45. To start test, the computer screen prompts the operator to press the "S" key on the computer keyboard. At this point, the computer assumes control of testing and the operator may observe the test being performed.

46. The computer applies the desired load for 1 second followed by a 20 second recovery period. After the recovery period the software places the rheometer into the load mode and a 100 gram load is applied for 240 seconds. As the test proceeds and the beam is loaded, the operator can observe computer screen plots of load and deformation versus time.

47. During the 240 seconds of testing, an embedded window on the computer screen shows a digital indication of actual load and deflection.

48. After 240 seconds, the computer ceases the test and automatically places the rheometer in the zero load mode. The software prompts the operator to press the "P" key to obtain a printout of the load and deflection plots, if desired. The computer also prompts the operator to press esc at which time the software automatically calculates test results, creep stiffness (S) and slope (m) of log creep stiffness versus log time plots. Finally, the operator is once again prompted to press the "P" key on the computer keyboard to obtain a printed copy of test results.

49. Remove the tested beam specimen from the beam supports and replace it with the other beam to be tested. The operator should return to the main menu by pressing esc and again, selecting "Run Test." Testing of the second beam proceeds as before.

50. A skilled operator should be able to test two beams in less than 10 minutes.

51. All of the required reporting items are printed by the rheometer software. These are:

- temperature of the test bath 60 s after the test load is applied, nearest 0.1° C,
- date and time,
- file name of test data,
- name of operator,
- sample identification number,
- flags issued by software during test,
- correlation coefficient R^2 for log stiffness vs log time, nearest 0.000001, and
- anecdotal comments.

Report the following items for time intervals of 8, 15, 30, 60, 120, and 240 seconds:

- time beams placed in bath,
- loading time, nearest 0.1 second,
- load, nearest 0.1 mN,
- beam deflection, nearest 2 μm,
- measured creep stiffness, nearest 0.001 MPa,
- estimated creep stiffness, nearest 0.001 MPa,
- difference between measured and estimated stiffness, %
- estimated m-value, nearest 0.001.

53. The operator should clean the mold pieces and plastic strips using a suitable solvent such as mineral spirits. In all cases, the mold pieces and plastic strips should be wiped with a clean cloth so that no solvent residue remains.

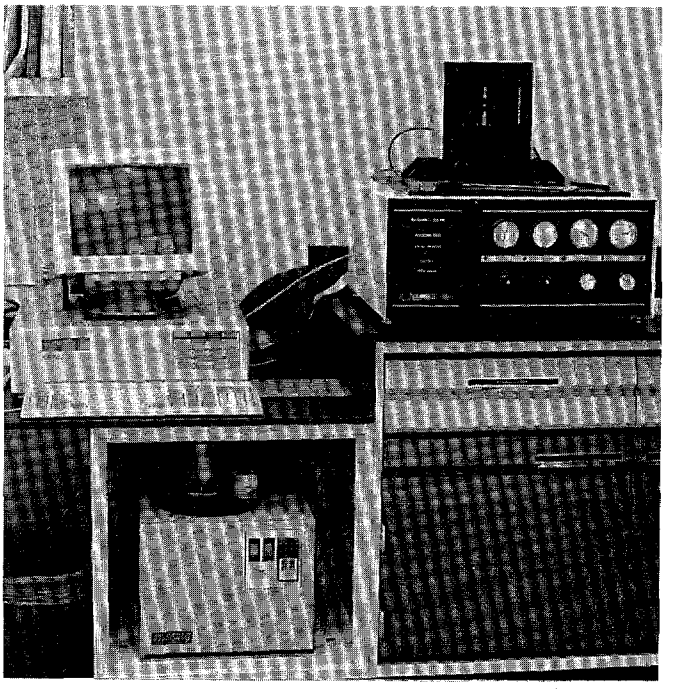


Figure 26. Bending Beam Rheometer

Direct Tension Tester

Test Method for Measuring Tensile Strain at Failure of Asphalt Binders at Low Temperatures Using a Direct Tension Apparatus

This document outlines the basic steps in measuring the tensile strain at failure of asphalt binders according to AASHTO Provisional Method TP3. It provides an illustrated overview, but it is not intended to replace the standard test method which contains detailed information pertaining to all aspects of the test.

1. This procedure outlines determination of tensile failure strain of asphalt binders. The apparatus (Figure 1) consists of a direct tension tester, a mechanical refrigeration unit to control temperature, a laser extensometer, and a computer that is used to control the tester and acquire data. The test is performed on binders that have been aged in a pressure aging vessel.

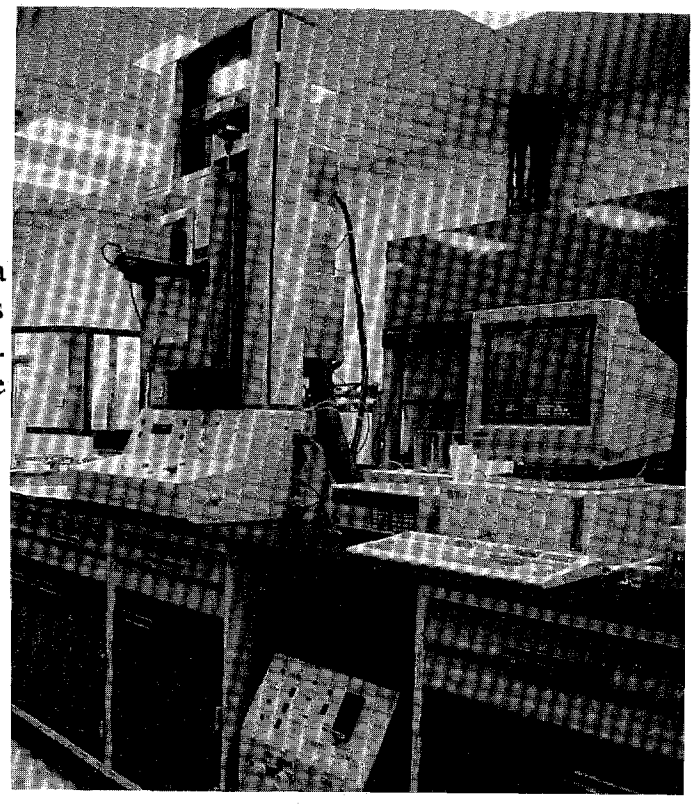
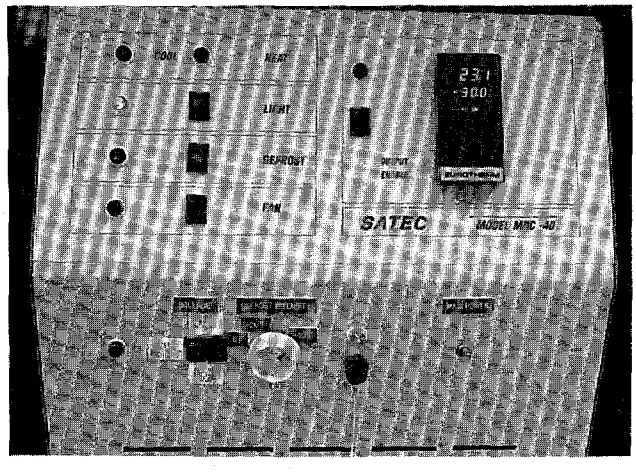


Figure 1. Direct Tension Apparatus

2. Turn on the mechanical refrigeration unit and set the desired test temperature. It should take no longer than 1 hour for the temperature to stabilize at the testing temperature.



3. Turn on the personal computer, the laser extensometer, and the direct tension testing machine. The laser device (controller shown in Figure 3) needs to be on at least 30 minutes prior to the first test.

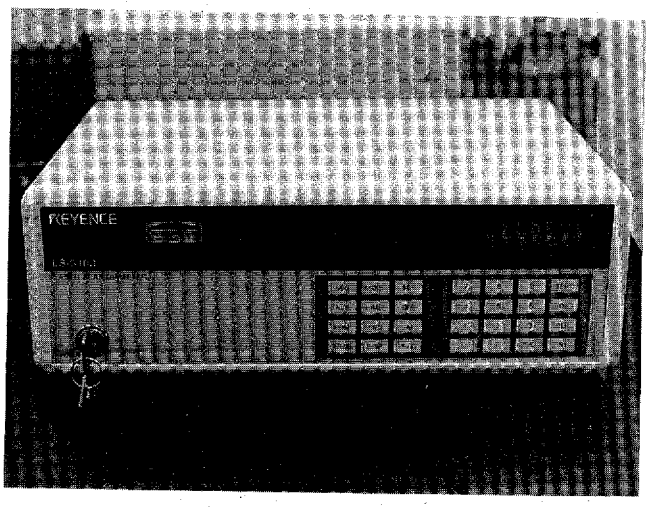


Figure 3. Laser Controller

4. Preheat the silicone rubber mold with inserts in oven at 135° C for 30 minutes. Upon removal from oven, place the mold on a 6 mm thick aluminum plate. Figure 4 shows the mold and inserts on the plate ready for filling.

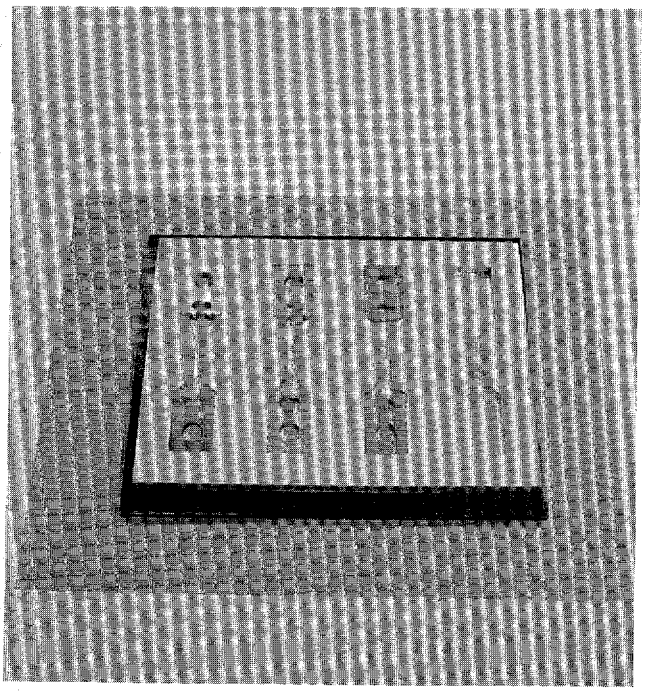


Figure 4. Silicone Rubber Mold with End Inserts and Aluminum Plate

5. Place a "3 ounce tin" containing the asphalt sample in the oven at approximately 135° C. The sample should be heated to achieve a viscosity of from 1 to 2 Pa·sec. Do not heat the sample above 150° C. While heating, the sample should be periodically stirred to remove any entrapped air. A single test will require 4 specimens, which is approximately 12 grams of total material.

6. Pour the asphalt sample into the preheated mold (Figure 5). At this point, the mold should remain on an aluminum plate on a level surface. The hot asphalt should be poured in a continuous stream in one pass starting at one end and proceeding to the opposite end until the mold is slightly overfilled. Repeat this procedure for the remaining three specimens in the mold.

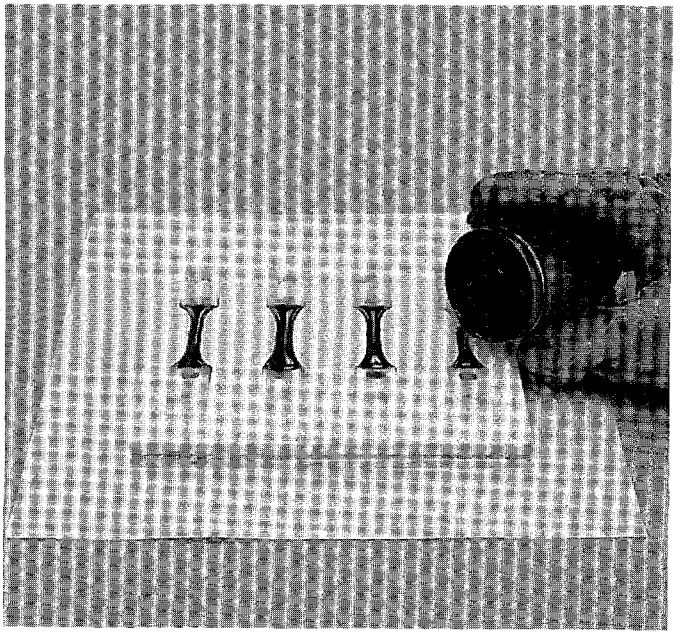


Figure 5. Pouring Sample to Make Specimens

7. The entire assembly should now be allowed to cool at ambient temperatures. The cooling process normally takes about 1 hour, which is the time necessary for the sample to cool to a condition at which it can be trimmed with no damage.

8. Trim the specimens using a hot spatula, putty knife, or other suitable tool such as a cheese plane (Figure 6). The tool should be wide enough to trim the specimen in one pass. Trim the specimen by placing the edge of the hot tool over one end insert and progressing to the other insert. Apply only slight pressure to avoid changing the thickness of the specimen and dislodging the inserts. To keep the trimming tool hot, place it over a bunsen burner or on a hot plate for a few seconds, as needed. After trimming, remove any asphalt from the holes or slots in the inserts.

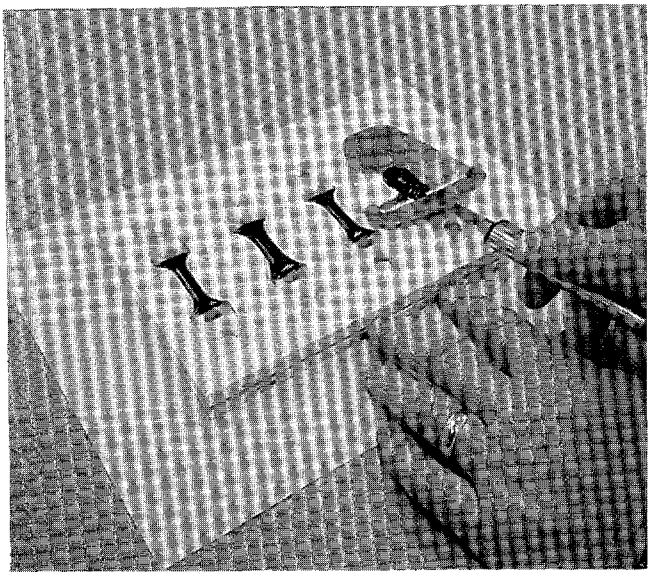


Figure 6. Specimen Trimming

9. Place the mold containing specimens in a freezer from 0° to -10° C for up to 15 minutes. This stiffens the specimens to facilitate demolding without deforming their shape.

10. Remove the mold containing specimens from the freezer. Place the assembly on a firm surface. Demold the specimens by gently bending the mold so that one side of the specimen is exposed (Figure 7). Grasp the specimen by one end insert and gently pull free from mold. Place the demolded specimen on a plastic plate (Figure 8). Repeat this procedure for the remaining three specimens.

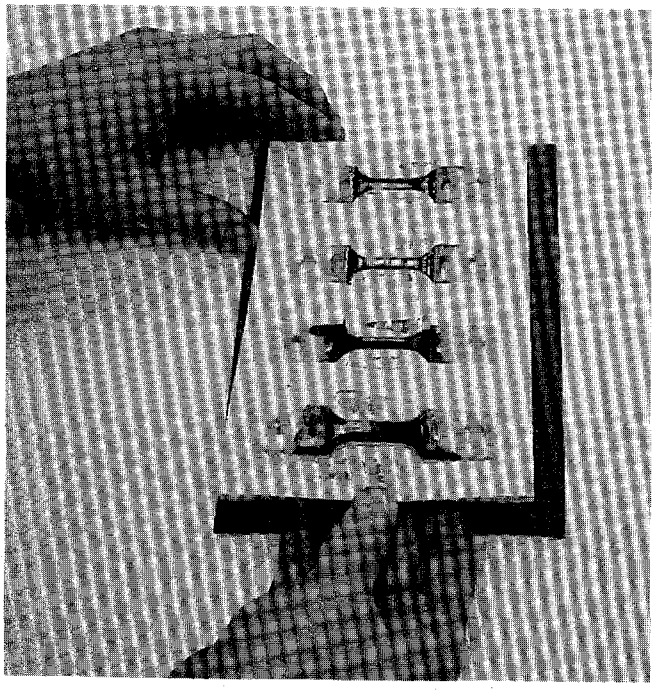


Figure 7. Demolding Specimens

11. Place the specimens still on the plastic plate into the temperature chamber. Use the small access port in the chamber door rather than opening the door (Figure 9). The plate containing the specimens should be placed on top of the shelf that is in close proximity to the temperature probe.

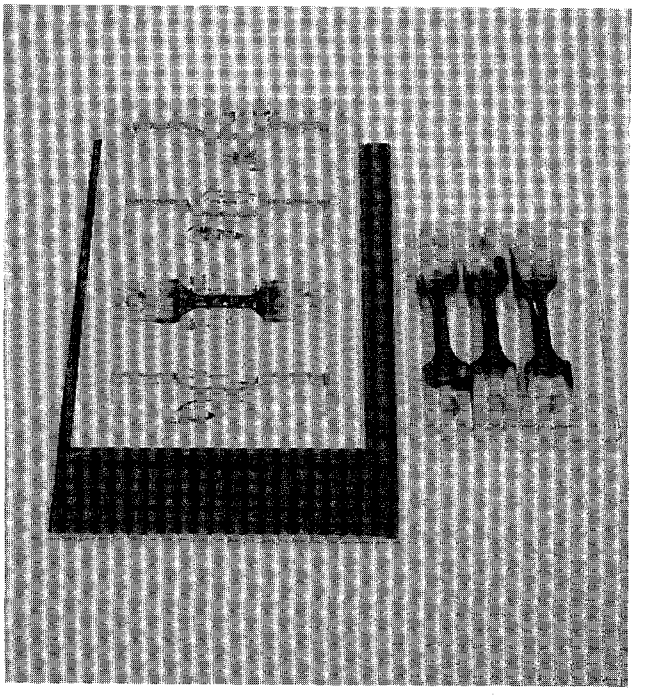


Figure 8. Specimens on Plastic Plate

12. At this point, the specimens should be conditioned for 1 hour ±10 minutes prior to testing. This interval, from 50 to 70 minutes, is established so that the operator can begin testing at 50 minutes and finish testing all four specimens before 70 minutes has elapsed.

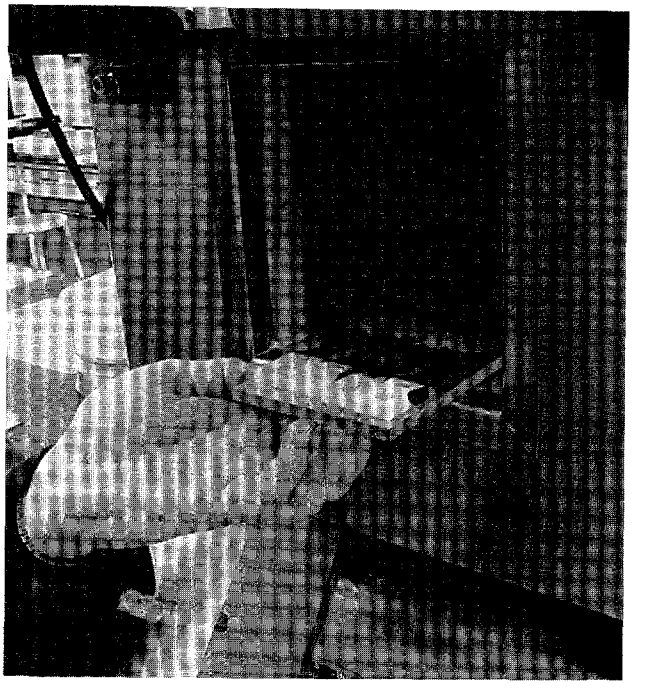


Figure 9. Specimens Going Into Temperature Chamber

13. During the conditioning period, the operator should verify testing parameters and prepare system software (if using a computer) for testing.

14. From the digital indicator on the testing device, the operator should verify that the machine is set to the proper mode to capture the desired test results: strain at failure, load at failure, and rate of elongation. An example of a digital display is shown in Figure 10. In this display, PS and PK indicate that the machine is set to capture peak strain and peak load, respectively. L2.....-0.1 is a real time display of load during the test. S4.....2.4536 is an indicator of the working range of peak elongation. The operator should consult the equipment manuals to ascertain how this verification step should be performed. The direct tension control panel is shown in Figure 11.

S4	2.4536	PS	.0000
L2	-0.1	PK	-0.1

Figure 10. Sample Digital Display on Direct Tension Tester

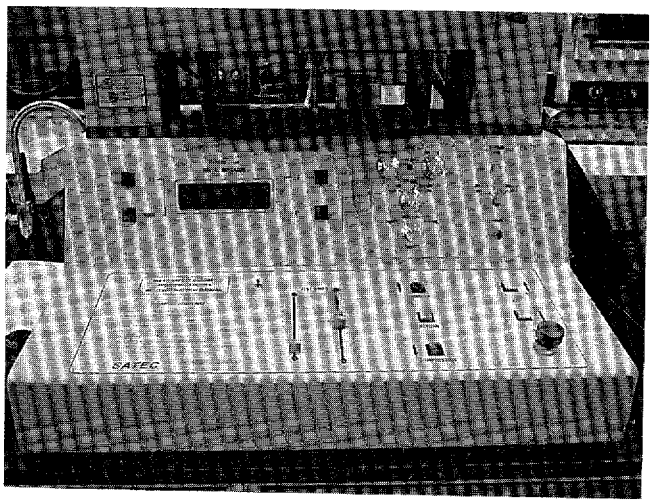


Figure 11. Direct Tension Tester Control Panel

15. Verify that the laser extensometer is operational by sliding a 3 x 20 cm strip of paper in front of the transmitting unit to partially block the plane of laser light. Observe the object positioning indicator on the front of the laser controller box. The positioning indicator is a row of lights that glow green when the paper is detected in the plane of laser light.

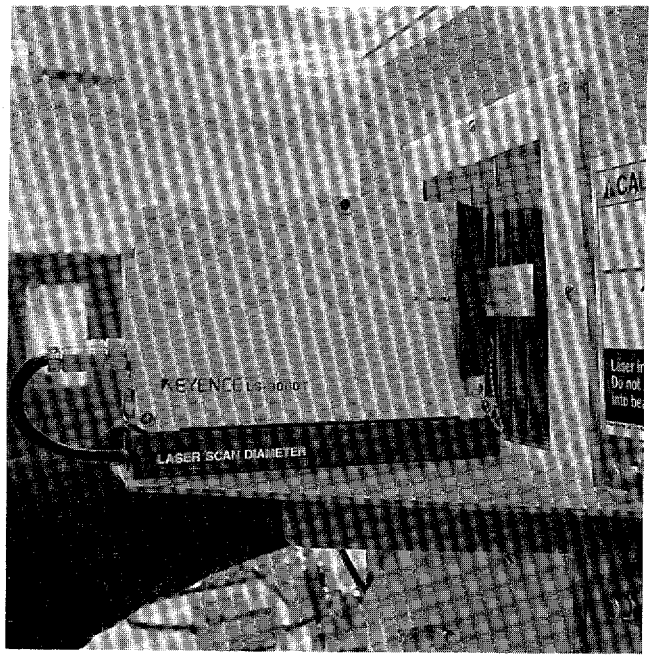


Figure 12. Sliding Paper in Front of Laser to Verify Operation

16. If data is captured by a computer, the operator should initialize the system software. From an input screen, the operator inputs routine, test-specific items such as operator name, sample identification number, test temperature, pressure aging parameters, etc. A skilled operator should be able to verify loading rate, testing parameters, and input test-specific information in less than 10 minutes.

17. At about 50 minutes after the specimens were placed in the chamber, they are ready to test. By reaching through the access hole and wearing rubber gloves, gently grasp a specimen by one of the inserts. Mount the specimen by slipping the hole of this insert over the top grip. Place a foam "doughnut" over the top pin to keep the specimen from being dislodged by the vigorous air flow (Figure 13). Position the bottom insert over the bottom grip. If the grip is out of position and does not align with the hole in the insert, use the testing machine "jog" contol (in fine adjustment) to reposition the grips. The jog control is simply a means to reposition the frame crosshead, and thus the grips, without applying load. Once the distance between the grips is established using this procedure, the "return" function will be used to precisely return the grips to the same position after each test.

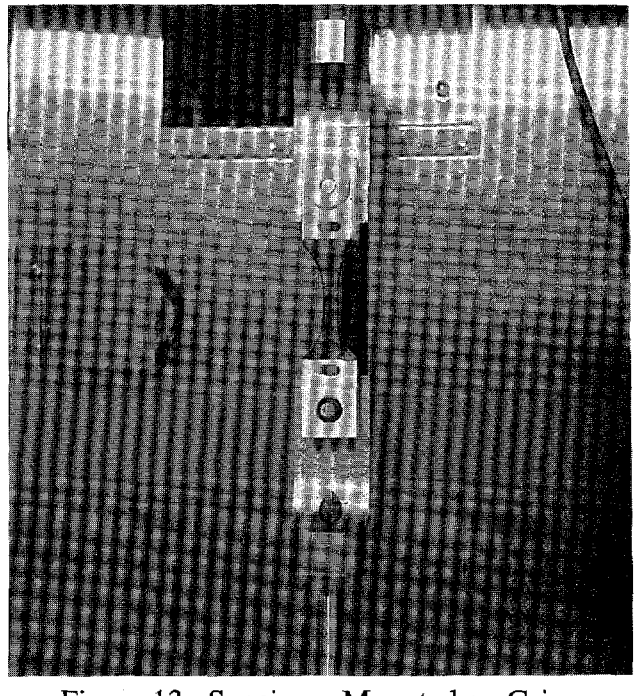


Figure 13. Specimen Mounted on Grips

18. Immediately prior to testing the operator should zero the load, peak load, and strain on the digital display. Zero the load by finely adjusting the load control knobs on the device control panel (Figure 14) while observing the digital display. The peak load is zeroed by pressing the clear button. Strain is zeroed by pressing a button or combination of buttons.

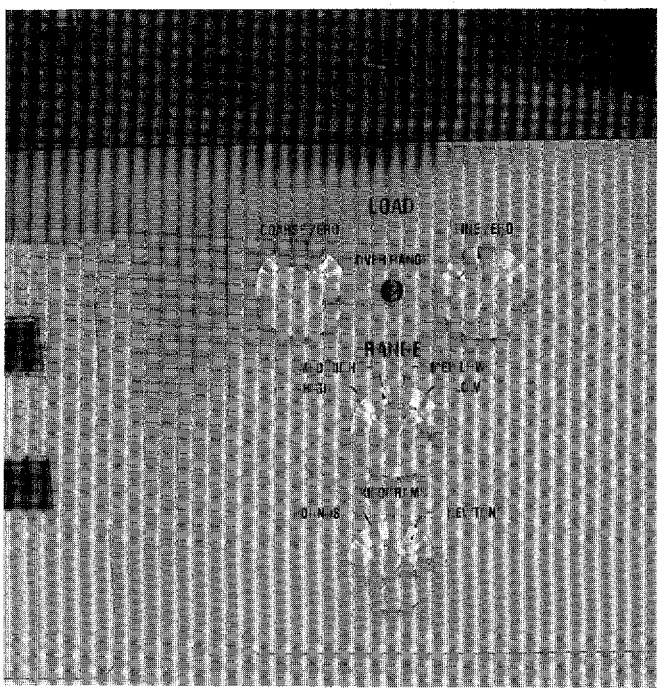


Figure 14. Load Control Adjustments

19. The laser extensometer should be zeroed by pressing the zero button on the panel of the laser controller box (Figure 15).

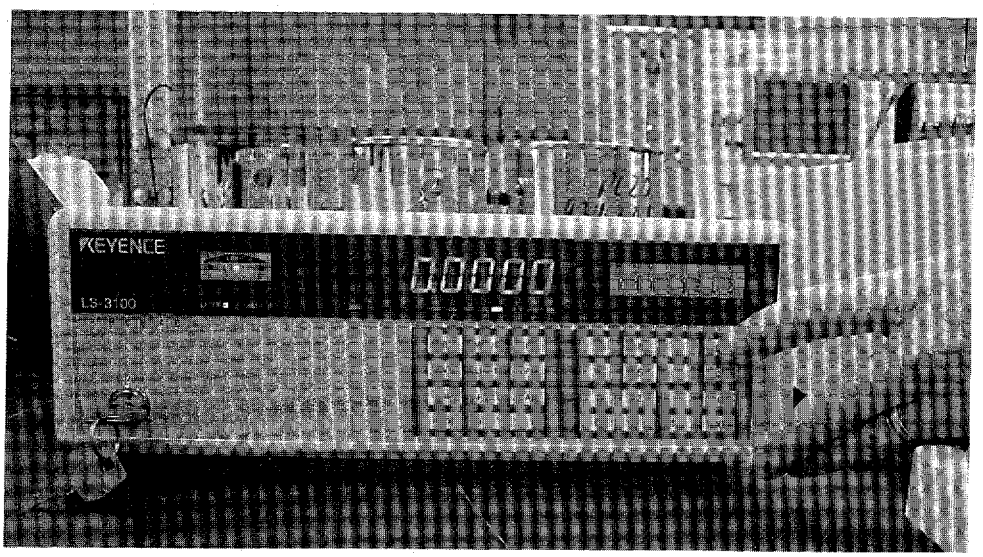


Figure 15. Zeroing Laser Extensometer

20. The operator should verify the chamber temperature by observing the digital display on the front of the mechanical refrigeration unit (Figure 16).

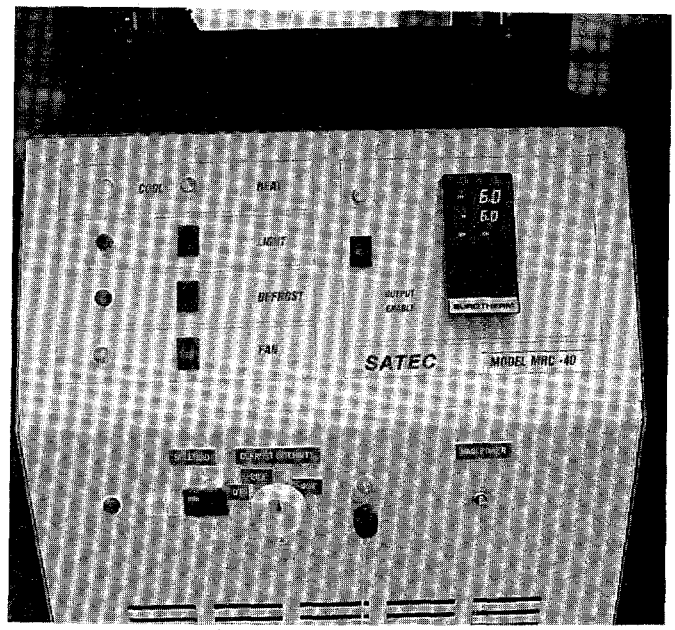


Figure 16. Mechanical Refrigeration Unit Showing Digital Temperature Display

21. If a computer is being used to acquire test results, the control program should be initialized.

22. To begin the test, press the "Tension" button on the machine control panel (Figure 17).

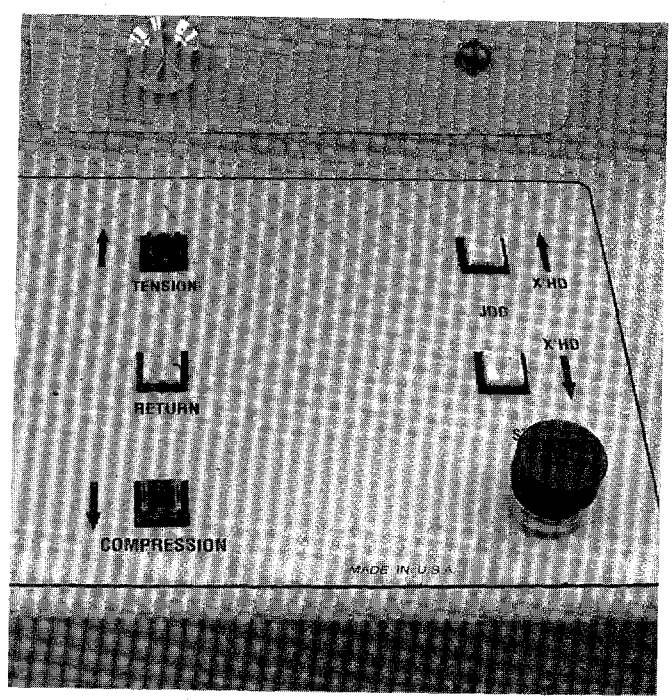


Figure 17. Control Panel with Tension Button

23. The test is complete when the specimen breaks. A break is considered valid when the specimen fractures within the narrow center portion (Figure 18).

24. From the digital display, record the strain at failure and peak load. If a computer is used to capture results, use the system software to store results.

25. Immediately proceed with testing the remaining three specimens using the aforementioned procedure. A skilled operator should be able to test the four specimens in less than 10 minutes.

26. After the fourth specimen has been tested, compute the average strain at failure and peak load. If a computer is used, system software computes these values automatically and operator only needs to print the test results.

27. The following items are reported:

- sample identification
- date and time of test
- test temperature, nearest 0.1° C,
- rate of elongation, nearest 0.01 mm/min,
- average failure strain, nearest 0.01 percent,
- average failure stress, nearest 0.01 MPa,
- average peak load, nearest N, and
- type of break observed (brittle, brittle-ductile, or no break).

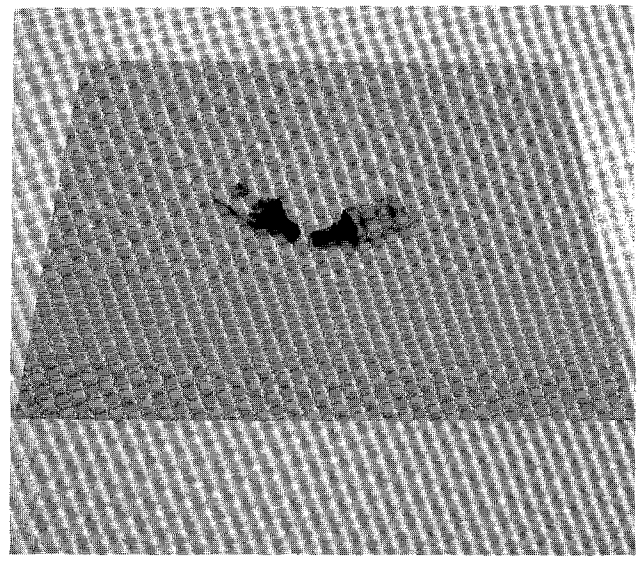


Figure 18. Correct Specimen Failure Mode

28. At the conclusion of testing, the operator should clean the mold and inserts. The mold is cleaned by heating it in an oven at about 135° C for 5 minutes and wiping it with a clean, dry cloth. The inserts are cleaned by using a hot knife to remove the failed specimens and then soaking them in mineral spirits. After soaking, wipe off excess mineral spirits and remove any residue by washing the inserts in a mild detergent. Rinse and dry the inserts with a clean cloth or paper towel. Do not use trichloroethylene or acetone to clean the inserts since they will cause damage.

29. The procedure for verifying loading rate varies depending on whether a computer is used with the direct tension device. If a computer is not available, the best approach is to use a dial gage and stop watch to measure travel of the specimen grips over a known length of time. Loading rate is computed by dividing the distance traveled in mm by the time interval in minutes.

30. To verify the loading rate when a computer is used with the direct tension device, mount a "dummy" specimen on the specimen grips inside the testing chamber. A dummy specimen consists of an elastic band looped through the inside holes of two inserts. Activate a tension load by pressing the "Tension" button on the loading device. Observe the loading rate on the computer screen to verify that it is at the prescribed rate of 1 ±0.05 mm/min.

31. Device calibration and verification should be performed periodically. This includes calibration of the load cell, laser extensometer, and temperature detector. Calibration procedures are provided in the test method.

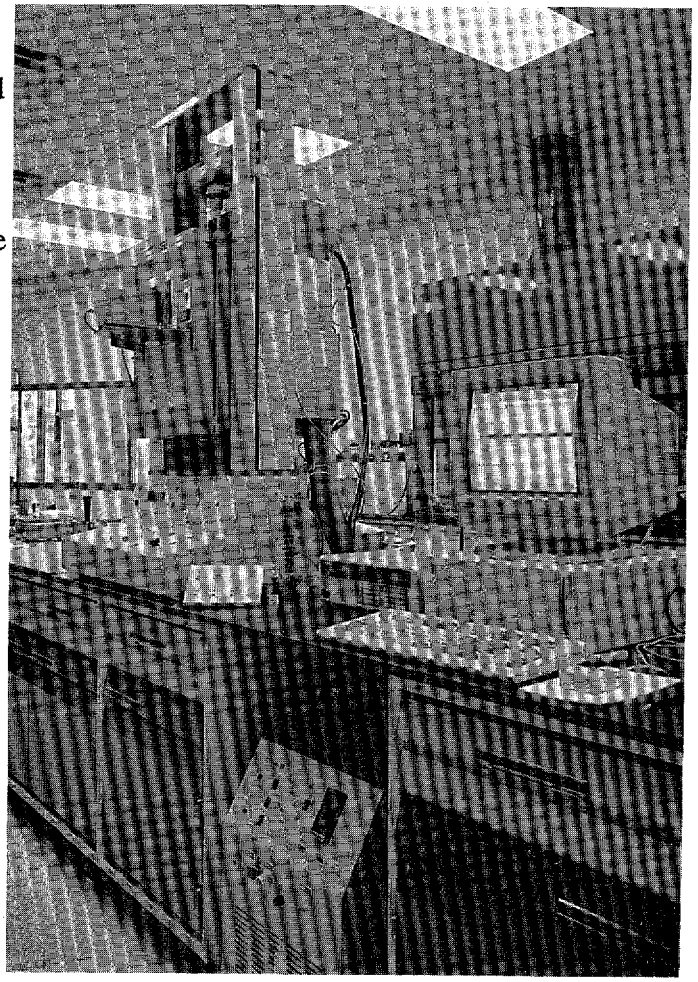


Figure 19. Direct Tension Testing Apparatus

APPENDIX A

AASHTO Provisional Standards for Asphalt Binders

MP1, PP1, PP5, PP6, TP1, TP3, TP5

September 1993

Standard Practice for
Grading or Verifying the Performance Grade
of an Asphalt Binder

AASHTO DESIGNATION: PP6[1]

1. Scope

1.1 This practice describes the testing required to determine the performance grade of an asphalt binder. It presents two approaches. In the first, the performance grade of an unknown asphalt binder is determined. In the second, the nominal performance grade of an asphalt binder is verified. It also provides an estimate of the time required to complete a single test sequence.

1.2 The values stated in SI units are to be regarded as standard.

1.3 This practice may involve hazardous materials, operations, and equipment. This practice does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this practice to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Reference Documents

2.1 AASHTO Documents:

MP1	Specification for Performance-Graded Asphalt Binder
PP1	Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)
T48	Method for Flash and Fire Points by Cleveland Open Cup
T179	Method for Effect of Heat and Air on Asphalt Materials (Thin-Film Oven Test)
T240	Method for Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin Film Oven Test)
TP1	Method for Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR)
TP3	Method for Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT)
TP5	Method for Determining Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

2.2 ASTM Documents:

D4402 Method for Viscosity Determinations of Unfilled Asphalt Using the Brookfield Thermosel Apparatus

3. Summary of the Practice

3.1 The tank (as-received) sample of asphalt binder is tested to determine the flash point in °C (T48), viscosity at 135°C (D4402), shear modulus (G^*) and phase angle (δ) (TP5).

3.2 The asphalt binder is aged in the rolling thin film oven (T240) or the thin film oven (T179), and the residue is tested to determine the mass loss (T240 or T179) and the shear modulus (G^*) and phase angle (δ) (TP5).

Note 1 -- T240 is the recommended procedure. Modified asphalt binders may phase separate or form skims during conditioning with T179 (TFOT); the results from subsequent testing of this residue in TP5, TP1 and TP3

[1] *This standard is based on SHRP Product P002.*

may be distorted.

3.3 The residue from the rolling thin film oven or thin film oven is aged in the pressurized aging vessel (PP1) and this residue is tested to determine the shear modulus (G^*) and phase angle (δ) (TP5), creep stiffness (S) and slope, m, of the log creep stiffness versus log time relationship at 60 seconds (TP1), the failure strain in direct tension (TP3), as necessary, and physical hardening measured by creep stiffness (S) and slope, m, of the log creep stiffness versus log time relationship at 1 and 24 hours (TP1).

3.4 Based on these test results the asphalt binder is graded according to MP1.

4. Significance and Use - This practice describes the testing required for grading or verifying the performance grade of an asphalt binder according to MP1.

5. Estimated Time Necessary for Testing - For both grading and verification, if the analysis is started at the beginning of a morning work shift, all testing and analysis should be completed during the afternoon of the next day. This schedule provides the 20 hours needed for PAV conditioning. (It takes 1 additional day to collect the physical hardening data as 24 hours of conditioning are required at the lowest grading test temperature to determine the extent of physical hardening.) Of course, samples can be aged and analyzed in parallel, and the productivity of the laboratory increased. However, for the purpose of this document, analysis of a single asphalt binder will be discussed.

6. Test Procedure for Grading an Unknown Asphalt Binder

6.1 Prepare samples and test specimens using the procedures specified in the test methods performed. In the case where the grade of the asphalt binder is unknown, approximately 400 g of unaged asphalt binder is required to complete the tests with the necessary replicates.

6.2 Begin conditioning asphalt binder in the rolling thin film oven (RTFO) or thin film oven (TFO). Condition a sufficient amount of asphalt binder depending on the type and number of tests to be performed.

> Note 2 -- Two BBR beams requiring PAV aged material are needed at each test temperature. In addition, four DT specimens requiring PAV aged material may also be needed at each test temperature. A minimum of two test temperatures will be required. Approximately 200 g of RTFO or TFO residue will be required.

6.3 Perform the DSR test (TP5) on the original asphalt binder beginning at 58°C, and increase or decrease the test temperature at 6.0°C increments until a value for $G^*/\sin\delta \leq 1.00$ kPa is obtained. The highest test temperature where the value for $G^*/\sin\delta$ is ≥ 1.00 kPa determines the starting PG grade.

> Note 3 -- For example, if $G^*/\sin\delta$ is 0.6 kPa at 64°C and 1.2 kPa at 58°C, the starting asphalt binder grade is PG 58-.

6.4 Determine the flash point on a sample of original binder using T48. The flash point must be greater than 230°C to meet the requirements of MP1.

6.5 Determine the viscosity of the original asphalt binder at 135°C using ASTM D4402. The viscosity must not exceed 3 Pa·s to meet the requirements of MP1.

6.6 After the RTFO test (T240) or TFO test (T179) is complete, Determine the mass loss of the original asphalt binder. The mass loss must be $\leq$ 1.00 percent to meet the requirements of MP1.

6.7 If the original asphalt binder does not meet MP1 requirements for the tests in Sections 6.4 and 6.5, no further testing is required.

6.8 Perform the DSR test (TP5) on the RTFO or TFO residue at the test temperature used to determine the starting PG grade (Section 6.3) to confirm the high temperature grade of the asphalt binder (PG 46-, 52-, 58-, etc.). The value for $G^*/\sin\delta$ of the RTFO or TFO residue must be $\geq$ 2.20 kPa. Choose the lower performance grade in cases where the test values in Sections 6.3 and 6.8 give conflicting grades.

6.9 Age a sufficient quantity of RTFO or TFO residue in the PAV (PP1). Use an aging temperature of 90°C for starting grades PG 46-x and 52-x binders and 100°C for starting grades PG 58-x and higher. PAV temperature of 110°C is to be used in simulating desert environments.

> Note 4 -- Two BBR beams (requiring PAV aged material) are required at each test temperature. In addition, four DT samples may also be needed at each test temperature. A minimum of two test temperatures will be required.

6.10 Complete steps 6.1 through 6.9 during the first day of testing. This will allow further testing to begin on the second day, when PAV aging is complete.

6.11 At the conclusion of the PAV aging procedure, carefully remove the residue from the vessel, combine individual pans of the same aged asphalt binder, and prepare two bending beam specimens for each test temperature according to TP1. Retain sufficient residue to prepare four direct tension specimens for each test temperature if required.

6.12 Perform the DSR test (TP5) on the PAV residue beginning at a test temperature of 16 and 19°C, respectively, for starting grades PG 52 and 58, 22°C for starting grade PG 64, and 28°C for starting grade PG 70, unless there is other information to suggest the temperature at which $G^*\sin\delta$ is $\leq$ 5000 kPa. Decrease or increase the test temperature at 3.0°C increments until the value for $G^*\sin\delta$ exceeds 5000 kPa.

6.13 Determine the beginning test temperature for the BBR test (TP1) on the PAV residue from Table 1 of MP1 using the starting PG grade determined in Section 6.3 and the lowest temperature from Section 6.12 where the value for $G^*\sin\delta$ did not exceed 5000 kPa, unless there is other information to suggest the temperature at which the stiffness is $\leq$ 300.0 MPa, and the slope m is $\geq$ 0.300.

6.14 Test pairs of BBR specimens according to TP1 beginning at the test temperature selected in Section 6.13 and increasing at 6.0°C increments, until a stiffness and slope meeting the requirements of MP1 are obtained. Test fresh BBR specimens at each temperature. Determine the extent of physical hardening, report the creep stiffness (S) and the slope, m, of the log creep stiffness versus log time relationship measured at 1 and 24 hours. Prepare two sets each of two BBR specimens of original asphalt binder. Test one set after 1 hour of conditioning and the second set after 24 hours of conditioning. The latter is conducted at the lowest grading test temperature (T_{min} + 10°C).

6.15 Certain asphalt binders may satisfy the MP1 slope requirement at substantially lower temperatures than they satisfy the stiffness requirement. If the creep stiffness is between 300 and 600 MPa at a test temperature at which the slope, m, is $\geq$ 0.300, it may be possible to satisfy the DT (TP3) failure strain requirement in lieu of the creep stiffness requirement. Test four DT specimens according to TP3 at the test temperature at which $m \geq 0.300$, and determine if the failure strain is $\geq$ 1.0 percent.

6.16 If the failure strain is < 1.0 percent, test additional sets of four DT specimens, increasing the test temperature in 6.0°C increments, until a failure strain $\geq$ 1.0 percent is obtained. Determine the test temperature at which m $\geq$ 0.300 and the creep stiffness is $\leq$ 300.0 MPa, or, if the creep stiffness is between 300.0 and 600.0 MPa, the failure strain is $\geq$ 1.0 percent.

6.17 Using the results of steps 6.12 through 6.16, determine the final grade of the asphalt binder.

7. Test Procedure for Verifying the Nominal Grade of An Asphalt Binder

7.1 Prepare samples and test specimens using the procedures specified in the test methods performed. In the case

where the grade of the asphalt binder is being verified, approximately 250 g of unaged asphalt binder is required to complete the tests with the necessary replicates.

7.2 Begin conditioning asphalt binder in the rolling thin film oven (RTFO) or thin film oven (TFO). Condition a sufficient amount of asphalt binder depending on the type and number of tests to be performed.

Note 5 -- Two BBR beams requiring PAV aged material are needed. In addition, four DT specimens requiring PAV aged material may also be required. Only one test temperature will be required. Approximately 100 g of RTFO or TFO, residue will be required.

7.3 Perform the DSR test (TP5) on the original asphalt binder at the test temperature indicated by the high temperature grading designation. For example, test a PG 70-16 asphalt binder at 70°C. The value for $G^*/\sin\delta$ must be $\geq$ 1.0 kPa to meet the requirements of MP1.

Note 6 -- This step verifies the starting PG grade. For example, if $G^*/\sin\delta \geq 1.00$ kPa at 58°C, the high temperature grading designation for the asphalt binder is PG 58-.

Note 7 -- If the asphalt binder fails to meet the requirements of MP1 for the grade that it has been designated, it may be treated as a binder of unknown grade and tested according to Section 6.

7.4 Determine the flash point on a sample of original binder using T48. The flash point must be greater than 230°C to meet the requirements of MP1.

7.5 Determine the viscosity of the original asphalt binder at 135°C using ASTM D4402. The viscosity must not exceed 3 Pa·s to meet the requirements of MP1.

7.6 After the RTFO test (T240) or TFO test (T179) is complete, determine the mass loss of the original asphalt binder. The mass loss must be $\leq$ 1.00 percent to meet the requirements of MP1.

7.7 If the original asphalt binder does not meet MP1 requirements for any of the tests performed in Sections 7.4, 7.5 or 7.6, no further testing is required.

7.8 To verify the high temperature properties of the binder, perform the DSR test (TP5) on the RTFO or TFO residue at the test temperature indicated by the high temperature grading designation. For example, test a PG 70-16 asphalt binder at 70°C. The value for $G^*/\sin\delta$ of the RTFO residue must be $\geq$ 2.20 kPa to meet the requirements of MP1. (Note 7)

7.9 Age a sufficient quantity of RTFO or TFO residue in the PAV (PP1). Use an aging temperature of 90°C for binders having a high temperature grading designation of PG 46-x or PG 52-x, 100°C for binders having high temperature grading designations of PG 58-x and higher. PAV temperature of 110°C is used in simulating desert environments.

Note 8 -- Two BBR beams (requiring PAV aged material) are required. In addition, four DT samples may also be needed requiring about 60 g of PAV aged asphalt binder.

7.10 Complete steps 7.1 through 7.9 during the first day of testing. This will allow further testing to begin on the second day, when PAV aging is complete.

7.11 At the conclusion of the PAV aging procedure, carefully remove the residue from the vessel, combine individual pans of the same aged asphalt binder, and prepare two bending beam specimens according to TP1. Retain sufficient residue to prepare four direct tension specimens if required.

7.12 Perform the DSR test (TP5) on the PAV residue at the test temperature specified in Table 1 of MP1 indicated for the high temperature and low temperature grading designation of the binder being verified. For example, test a PG 64-40 asphalt binder at 16°C. The value for $G^*\sin\delta$ must not exceed 5000 kPa to meet the requirements of MP1. (Note 7)

7.13 Test two BBR specimens according to TP1 at the test temperature specified in Table 1 of MP1 indicated for the high temperature and low temperature grading designation of the binder being verified. For example, test a PG 58-28 asphalt binder at -18°C. The value of the slope m must be $\geq$ 0.300 to meet the requirements of MP1. Certain asphalt binders may satisfy the BBR slope requirement at substantially lower temperatures than they satisfy the BBR stiffness requirement. (Note 7)

7.14 Determine the extent of physical hardening, report the creep stiffness (S) and the slope, m, of the log creep stiffness versus log time relationship measured at 1 (conducted above in section 7.13) and 24 hours. Prepare two BBR specimens of original asphalt binder. Condition and test the specimens at the test temperature, selected from Table 1 of MP1, appropriate for the performance grade of the asphalt binder. For example, if the binder grade being verified is a PG 64-34 asphalt binder, condition and test the BBR specimens at -24°C. Test one set after 1 hour of conditioning and the second set after 24 hours of conditioning.

7.15 If the creep stiffness is between 300.0 and 600.0 MPa and the slope m is $\geq$ 0.300 at the test temperature, it may be possible to satisfy the DT failure strain requirement in lieu of the creep stiffness requirement. Test four DT samples according to TP3 at the same test temperature used to test the BBR specimens. The failure strain must be $\geq$ 1.0 percent to meet the requirements of MP1. (Note 7)

8. Report

8.1 If the grade of the asphalt binder tested is determined, report the results of all tests performed and the high temperature grading designation determined followed by the low temperature designation (for example: PG 52-34).

8.2 If the grade of an asphalt binder is verified, report the results of all tests performed and if the binder meets the requirements of MP1.

9. Keywords - asphalt binder, performance grading

Standard Practice for
Accelerated Aging of Asphalt Binder
Using a Pressurized Aging Vessel (PAV)

AASHTO Designation: PP1[1]

1. Scope

1.1 This practice covers the accelerated aging (oxidation) of asphalt binders by means of pressurized air and elevated temperature. The test method is intended to simulate in-service oxidative aging of asphalt binders and is intended for use with residue from T240 (RTFOT) or T179 (TFOT).

> Note 1 -- T240 is the recommended procedure. Modified asphalt binders may phase separate or form skims during conditioning with T179 (TFOT); the results from subsequent testing of this residue in TP5, TP1, and TP3 may be distorted.

1.2 The aging of asphalt binders during service is affected by mixture-associated variables such as the volumetric proportions of the mix, the permeability of the mix, properties of the aggregates, and possibly other factors. This test is intended to provide an evaluation of the relative resistance of different asphalt binders to oxidative aging at selected temperatures and cannot account for mixture variables.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. Reference Documents

2.1 AASHTO Standards:

M231 Standard Specification for Standard Masses and Balances Used in the Testing of Highway Materials
MP1 Proposed Standard Specification for Performance-Graded Asphalt Binder
T179 Test Method for Effect of Heat and Air on Asphalt Materials (Thin-Film Oven Test)
T240 Test Method for Effect of Heat and Air on Rolling Film of Asphalt (Rolling Thin-Film Oven Test)

2.2 ASTM Standards:

E220 Method for Calibration of Thermocouples by Comparison Techniques

2.3 DIN Standards:

43760

3. Terminology

3.1 Definitions

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[1] *This standard is based on SHRP Product 1002.*

3.1.1 asphalt binder, n — an asphalt-based cement that is produced from petroleum residue either with or without the addition of non-particulate organic modifiers.

3.1.2 in-service, adj — refers to aging of the asphalt binder that occurs in the pavement as a result of the combined effects of time, traffic and the environment.

4. Summary of Test Method

4.1 Asphalt binder is first aged using T240 (RTFOT) or T179 (TFOT). A specified thickness of residue, from the RTFOT or TFOT, is then placed (or left) in standard TFOT (T179) stainless steel pans and aged at the specified aging temperature for 20 hours in a vessel pressurized with air to 2.10 MPa. Aging temperature is selected according to the grade of the asphalt binder.

5. Significance and Use

5.1 This method is designed to simulate the in-service oxidative aging that occurs in asphalt binders during pavement service. Residue from this test may be used to estimate the physical or chemical properties of asphalt binders after five to ten years of in-service aging in the field.

5.2 Asphalt binders aged using PP1 are used to determine specification properties in accordance with MP1. The asphalt binder is aged with the RTFO or TFO test prior to this conditioning step. Tank asphalt binders, as well as RTFOT or TFOT, and residue from this test are used to determine specification properties in accordance with MP1

5.3 For asphalt binders of different grades or from different sources, there is no unique correlation between the aging time and temperature in this test and in-service pavement age and temperature. Therefore, for a given set of in-service climatic conditions, it is not possible to select a single PAV aging time and temperature that will predict the properties of all asphalt binders after a specific set of in-service exposure conditions.

5.4 The relative degree of hardening of different asphalt binders varies at different aging temperatures in the PAV. Therefore, two asphalt binders may age similarly at one temperature, but age differently at another temperature.

6. Apparatus

6.1 A test system consisting of a pressure vessel, pressure controlling devices, temperature controlling devices, pressure and temperature measuring devices, and a temperature recording device (Figure 1).

6.1.1 Pressure Vessel - A stainless steel pressure vessel designed to operate at 2.1 ± 0.1 MPa between 90 and 110°C with interior dimensions no greater than 200 mm in diameter and 215 mm in height (adequate to hold 10 TFOT pans and pan holder). The pressure vessel shall contain a pan holder capable of holding ten TFOT stainless steel pans in a horizontal (level) position such that the asphalt binder film thickness in the bottom of the pans does not vary by more than 0.1 mm across any diameter of the pan. The holder shall be designed for easy insertion and removal from the vessel when the holder, pans, and asphalt binder are at the test temperature. A schematic showing the vessel, pan holder and pans and specifying dimensional requirements is shown in Figure 2.

> Note 2 -- The vessel may be a separate unit to be placed in a forced draft oven for conditioning the asphalt binders or an integral part of the temperature control system (for example by direct heating of the vessel or by surrounding the vessel with a permanently affixed heating unit, forced air oven, or liquid bath).

6.1.2 Pressure Controlling Devices

6.1.2.1 A pressure release valve that prevents pressure in the vessel from exceeding 2.2 MPa during the aging procedure.

6.1.2.2 A pressure regulator capable of controlling the pressure within the vessel to ± 1 percent, and with a capacity adequate to reduce the pressure from the source of compressed air so that the pressure within the loaded pressure vessel is maintained at 2.1 ± 0.1 MPa during the test.

6.1.2.3 A slow release bleed valve that allows the pressure in the vessel at the completion of the conditioning procedure to be reduced at an approximately linear rate from 2.1 MPa to local atmospheric pressure within 9 ± 1 minutes.

6.1.3 Temperature Controlling Devices - A temperature control device as described in Section 6.1.3.1 or 6.1.3.2 for maintaining the temperature during the aging procedure at all points within the pressure vessel at the aging temperature ± 0.5°C and a digital proportional controller for maintaining the specified temperature control.

6.1.3.1 A forced-draft oven or fluid bath capable of (1) bringing the loaded unpressurized vessel to the desired conditioning temperature ± 0.5°C, as recorded by the Resistance Thermal Detector (RTD) inside the vessel, within 2 hrs., and (2) maintaining the temperature at all points within the pressure vessel at the aging temperature ± 0.5°C. The oven shall have sufficiently large interior dimensions to allow forced air to freely circulate within the oven and around the pressure vessel when the vessel is placed in the oven. The oven shall contain a stand or shelf which supports the loaded pressure vessel in a level position above the lower surface of the oven (i.e. maintains the film thickness in the aging pans within the specified tolerance).

6.1.3.2 A pressure vessel with integral temperature control system that is capable of (1) bringing the loaded pressure vessel to the desired conditioning temperature ± 0.5°C within 2 hrs., as recorded by the RTD inside the loaded pressure vessel, and (2) maintaining the temperature at all points within the pressure vessel at the aging temperature ± 0.5°C.

6.1.4 Temperature and Pressure Measuring Devices:

6.1.4.1 A platinum RTD accurate to the nearest 0.1°C and meeting DIN Standard 43760 (Class A), or equal, for measuring temperature inside the pressure vessel. The RTD shall be calibrated as an integral unit with its respective meter or electronic circuitry.

> Note 3 -- The RTD or thermistor and its meter may be calibrated by the manufacturer or a commercial vendor. Verification can be obtained by comparing the output from the RTD with a NIST traceable ASTM 94C mercury in glass thermometer in accordance with ASTM E220. A stirred fluid bath is suitable for calibrating the thermal detector. Select a partial immersion mercury-in-glass thermometer with an appropriate range and place the thermal detector and the thermometer in the stirred water bath. Fasten the detector to the glass thermometer with a rubber band or rubber O-ring. Allow the bath, detector, and thermometer to come to thermal equilibrium and record the temperature of the glass thermometer and the readout from the thermal detector. The temperature in the bath shall not change by more than 0.1°C per minute during the calibration process.

6.1.4.2 Temperature Recording Device - A strip chart recorder or other data acquisition system capable of recording temperature throughout the test to 0.1°C. As an alternative, an electronic device capable of reporting maximum and minimum temperatures (accurate to ± 0.1°C) may be used. In this case if the test temperature varies by more than ± 0.5°C of the conditioning temperature during the 20-hour period, the test shall be declared invalid.

6.1.4.3 A pressure gauge capable of measuring the pressure in the pressure vessel to within ± 1 percent during the test.

6.2 Stainless Steel Pans - Ten standard stainless steel TFOT pans meeting the requirements of T179.

6.3 Balance - A balance conforming to the requirements of M231, Class G2.

7. Materials - Commercial bottled air or equivalent.

8. Hazards - Use standard laboratory safety procedures in handling the hot asphalt binder when preparing test specimens and removing the residue from the pressure vessel. Use special precaution when lifting the pressure vessel.

9. Calibration and Standardization

9.1 Temperature Detector - Verify the calibration of the RTD to 0.1°C at least every 6 months using a calibrated thermometer.

9.2 Pressure gauge - Calibrate the pressure gauge to an accuracy of 1 percent at least every 6 months.

Note 4 -- The pressure gauge is usually calibrated by the manufacturer or a commercial calibration service. Verification of the continued stability of the pressure gauge within the specified requirements should be done periodically by checking against another certified pressure measurement device.

10. Procedure

10.1 Condition the asphalt binder and determine the mass change during conditioning in accordance with T240 (RTFOT) or T179 (TFOT).

10.2 Combine the hot residue from the RTFOT into a single container, stir to blend, then transfer into (or leave in) TFOT pans for PAV conditioning or allow the hot residue in the container to cool to room temperature and cover and store at room temperature for PAV conditioning at a later date. If conditioned asphalt binder is allowed to cool to room temperature, heat it until it is sufficiently fluid to pour and stir it before pouring it into the TFOT pans. To remove asphalt binder from RTFOT bottles, scrapping of the bottles is allowed, to assure sufficient material is obtained for later testing. Scrapping is not currently allowed in T240. If scrapping is used, report with the test results.

10.3 Place the pan holder inside the pressure vessel. If an oven is used, place the pressure vessel inside the oven. If an integrated temperature control pressure vessel is used, turn on the heater. Select an aging temperature and preheat the pressure vessel to the aging temperature selected.

Note 5 -- If conditioning asphalt binders for conformance to MP1, select the appropriate aging temperature from Table 1 of MP1.

Note 6 -- Preheating the vessel 10 to 15°C above the conditioning temperature can be used to reduce the drop in PAV temperature during the loading process and minimize the time required to stabilize the system, after loading, to attain the required temperature.

Note 7 -- Aging temperature in the PAV is selected to account for different climatic regions. Temperatures in excess of approximately 115°C can change the chemistry of asphalt binders aged in accelerated tests and should be avoided.

10.4 Place the TFOT pan on a balance and add 50 $\pm$ 0.5 gram mass of asphalt binder to the pan. This will yield approximately a 3.2 mm thick film of asphalt binder.

Note 8 -- The mass change is not measured as part of this procedure. Mass change is not meaningful because the asphalt binder absorbs air as a result of pressurization. Any gain in mass as a result of oxidation is masked by air absorbed by the asphalt binder as a result of the pressurization.

10.5 If the vessel is preheated to other than the desired aging temperature, reset the temperature control on the heating device to the aging temperature.

10.6 Place the filled pans in the pan holder. (Pans containing asphalt binders from different sources and grades

may be placed in the pressure vessel during a single test.) Place the panholder with filled pans inside the pressure vessel and close the pressure vessel.

10.7 If an oven is used, place the loaded and closed pressure vessel in the oven.

10.8 Connect the temperature transducer line and the air pressure supply line to the loaded pressure vessel's external connections.

10.9 Perform the operations described in Sections 10.5 to 10.8 as quickly as possible to avoid cooling of the vessel and pan holder.

10.10 Wait until the temperature inside the pressure vessel is within 2°C of the aging temperature, apply an air pressure of 2.1 ± 0.1 MPa and then start timing the test. If the temperature inside the pressure vessel in not attained within two hours of loading discontinue the test.

Note 9 -- Pressures in excess of 2.1 MPa do not substantially increase the rate of aging. Therefore, higher pressures are not warranted.

Note 10 -- Once pressurized, the temperature inside the pressure vessel will equilibrate rapidly. The time under pressure, not to include any preheating time at ambient pressure, is the aging time. Relatively little aging occurs at ambient pressure during the time that the vessel is being reheated to the test temperature, given that asphalt binder residue under test has been exposed to 163°C in the RTFOT.

10.11 Maintain the temperature and air pressure inside the pressure vessel for 20 hours ± 10 minutes.

10.12 At the end of the 20-hour test period slowly begin reducing the internal pressure of the PAV, using the air pressure bleed valve. Adjust the bleed valve to an opening that requires 9 ± 1 minutes to equalize the internal and external pressures on the PAV, thus avoiding excessive bubbling and foaming of the asphalt binder. During this process it may be necessary to adjust the setting of the needle valve as the pressure drops in order to maintain an approximate linear rate of pressure decrease. Do not include the pressure release and equalization time as part of the 20 hour aging period.

10.13 If the temperature indicated by the temperature recording device falls above or below the target aging temperature ± 0.5°C for more than 10 minutes during the 20 hour aging period, declare the test invalid and discard the material.

10.14 Remove the pan holder and pans from the PAV, and place in an oven set at 163°C. Heat until sufficiently fluid to pour. Stir gently to assist in the removal of air bubbles.

Note 11 -- Minimum pouring temperatures that produce a consistency equivalent to that of SAE 10W30 motor oil (readily pours but not overly fluid) at room temperature are recommended. Heating unaged asphalt binders to temperatures above 135°C should be avoided, however, with some modified asphalts or heavily aged binders pouring temperatures above 135°C may be required. PAV residue are allowed to be heated in the TFOT pans to 163°C and stirred to remove air bubbles. In all cases heating time should be minimized. These precautions will help avoid oxidative hardening and volatile loss that will harden the sample. During the heating process the sample should be covered and stirred occasionally to ensure homogeneity.

10.15 Remove pans from oven and pour the hot residue from the pans into a single container. If tests to determine the properties of the PAV residue are not performed immediately, cover the container and store it at room temperature for future testing.

11. Report

11.1 Report the following information.

11.1.1 sample identification

11.1.2 aging test temperature, nearest 0.5°C

11.1.3 maximum and minimum aging temperature recorded, nearest 0.1°C

11.1.4 total time during aging that temperature was outside the specified range, nearest minute

11.1.5 total aging time, hours and minutes

11.1.6 report the heating temperature and heating time if temperatures greater than 163 °C are required at any time during the handling of the material

12. Precision and Bias

12.1 Precision - The research required to develop precision estimates for tests performed on PAV residue has not been conducted.

12.2 Bias - The research required to establish the bias of tests performed on PAV residue has not been conducted.

13. Keywords - pressure aging, elevated temperature, PAV, pressure aging vessel, in-service aging, accelerated aging.

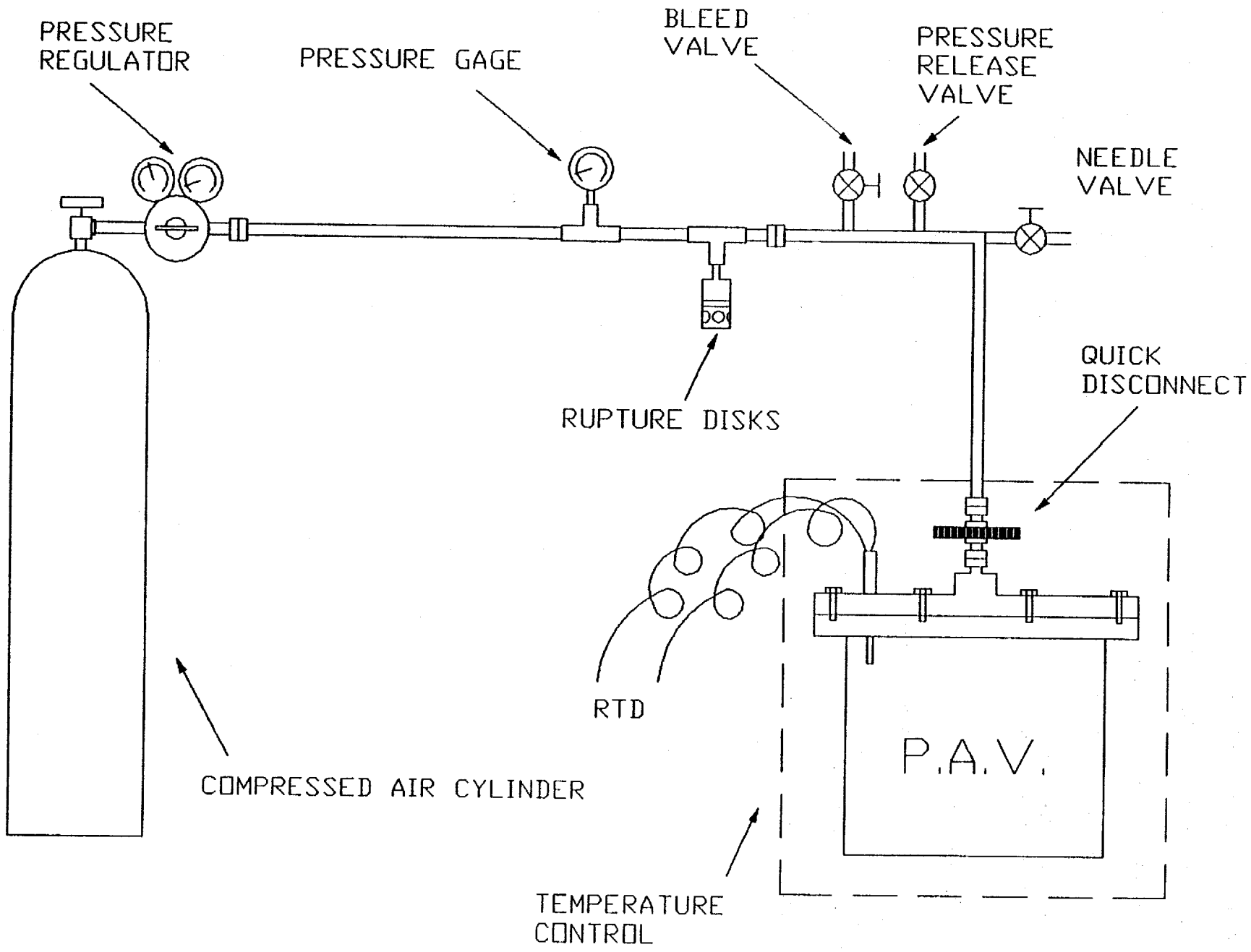
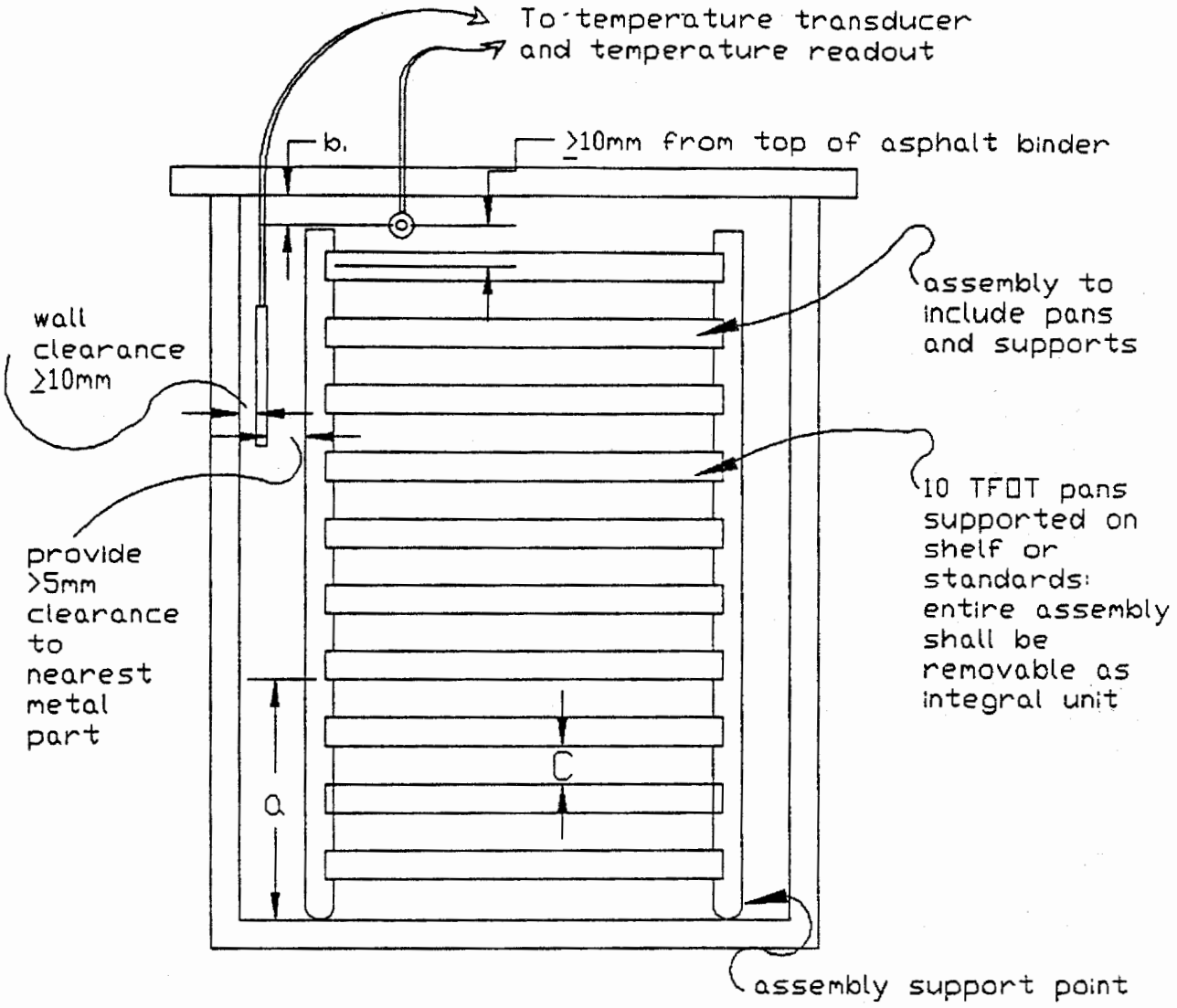


Figure 1: Typical Schematic of PAV Test System



Note 1: Distance "a" controls the levelness of the pans. The assembly shall be supported at 3 or 4 support points. The distance "a", measured from each assembly support point to the bottom of the pan (top of shelf or pan support point), shall be controlled to ±0.05mm. Provision shall be made to insure that the bottom of the vessel is leveled so that the thickness of the binder in the pans varries by no more than ±0.05 mm across the diameter of any pan.

Note 2: Disance b shall be such that any active portion of the temperature tranducer is ≥10mm from the top surface of the vessel.

Note 3: Distance c shall be ≥ 12mm.

Figure 2- Schematic showing location of pans and RTD within representative PAV

Standard Test Method for Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR)

AASHTO Designation: TP1[1]

1. Scope

1.1 This test method covers the determination of the flexural creep stiffness or compliance of asphalt binders by means of a bending beam rheometer. It is applicable to material having flexural creep stiffness values from 30 MPa to 1 GPa (creep compliance values in the range of 300 mPa^{-1} to 1 nPa^{-1}) and can be used with unaged material or with material aged using T240 (RTFOT), T179 (TFOT), or PP1 (PAV). The test apparatus is designed for testing within the temperature range from -40 to 25°C.

1.2 Test results are not valid for beams of asphalt binder that deflect more than 4 mm when tested in accordance with this method.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. Reference Documents

2.1 AASHTO Standards

- **MP1** Specification for Performance-Graded Asphalt Binder
- **PP1** Practice for Accelerated Oxidative Aging of Asphalt Binder Using Pressurized Air and Elevated Temperature (PAV)
- **T40** Practice for Sampling Bituminous Materials
- **T179** Test Method for Effect of Heat and Air on Asphalt Materials (Thin-Film Oven Test)
- **T240** Test Method for Effect of Heat and Air on Rolling Film of Asphalt (Rolling Thin-Film Oven Test)

2.2 ASTM Standards

- **C802** Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials
- **E1** Specification for ASTM Thermometers
- **E77** Standard Test Method for Inspection and Verification of Liquid-in-Glass Thermometers

2.3 DIN Standards

43760

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[1] *This standard is based on SHRP Product 1002.*

3. Terminology

3.1 Definitions

3.1.1 asphalt binder, n — an asphalt-based cement that is produced from petroleum residue either with or without the addition of non-particulate organic modifiers.

3.1.2 physical hardening, n — a time-dependent stiffening of asphalt binder that results from time-delayed increase in stiffness when the asphalt binder is stored at low temperatures.

3.2 Descriptions of Terms Specific to this Standard

3.2.1 flexural creep, n — a material characteristic determined by a test in which a simply-supported beam is loaded with a constant load at its midpoint and the deflection of the beam is measured with respect to loading time.

3.2.2 flexural creep stiffness, S(t) — ratio obtained by dividing the maximum bending stress in the beam by the maximum bending strain.

3.2.3 flexural creep compliance, D(t) — ratio obtained by dividing the maximum bending strain in the beam by the maximum bending stress. D(t) is the inverse of S(t). S(t) has been used historically in asphalt technology while D(t) is commonly used in studies of viscoelasticity.

3.2.4 *m* — absolute value of the slope of the logarithm of the stiffness curve versus the logarithm of time.

3.2.5 preload, n — load required to maintain positive contact between the beam and the loading shaft; 30 $\pm$ 5 mN.

3.2.6 initial seating load, n — load of 1-s duration required to seat the beam; 980 $\pm$ 50 mN.

3.2.7 test load, n — load of 240-s duration required to determine the stiffness of the material being tested; 980 $\pm$ 50 mN.

4. Summary of Test Method

4.1 The bending beam rheometer measures the mid-point deflection of a simply-supported prismatic beam of asphalt binder subjected to a constant load applied to the mid-point of the beam. The device operates only in the loading mode; recovery measurements are not obtained.

4.2 A test beam is placed in the controlled temperature fluid bath and loaded with a constant load for 240 seconds. The test load (980 $\pm$ 50 mN) and the mid-point deflection of the beam are monitored versus time using a computerized data acquisition system.

4.3 The maximum bending stress at the midpoint of the beam is calculated from the dimensions of the beam, the span length, and the load applied to the beam for loading times of 8, 15, 30, 60, 120, and 240 seconds. The maximum bending strain in the beam is calculated for the same loading times from the dimensions of the beam and the deflection of the beam. The stiffness of the beam for the loading times specified above is calculated by dividing the maximum stress by the maximum strain.

5. Significance and Use

5.1 The test temperature for this test is related to the temperature experienced by the pavement in the geographical area for which the asphalt binder is intended.

5.2 The flexural creep stiffness or flexural creep compliance, determined from this test, describes the low-temperature stress-strain-time response of asphalt binder at the test temperature within the linear viscoelastic response range.

5.3 The low-temperature thermal cracking performance of paving mixtures is related to the creep stiffness and the slope of the logarithm of the creep stiffness versus the logarithm of the time curve of the asphalt binder contained in the mix.

5.4 The creep stiffness and the slope of the logarithm of the stiffness versus the logarithm of the time curve are used as performance-based specification criteria for asphalt binders in accordance with MP1.

6. Interferences

6.1 Measurements wherein the beam deflection is greater than 4.0 mm are suspect. Strains in excess of this value may exceed the linear response of asphalt binders.

6.2 Measurements wherein the beam deflection is less than 0.08 mm are suspect. When the beam deflection is less than 0.08 mm the test system resolution may not be sufficient to produce reliable test results.

7. Apparatus

7.1 Bending Beam Rheometer (BBR) Test System - A bending beam rheometer (BBR) test system consisting of (1) a loading frame which permits the test beam, supports, and the lower part of the test frame to be submerged in a constant temperature fluid bath, (2) a controlled temperature liquid bath which maintains the test beam at the test temperature and provides a buoyant force to counterbalance the force resulting from the mass of the beam, and (3) a computer controlled automated data acquisition component.

7.1.1 Loading Frame - A frame consisting of a set of sample supports, a blunt-nosed shaft that applies the load to the midpoint of the test specimen, a load cell mounted on the loading shaft, a means for zeroing the load on the test specimen, a means for applying a constant load to the loading shaft, and a deflection measuring transducer attached to the loading shaft. A schematic of the device is shown in Figure 1.

7.1.1.1 Sample Supports - Sample supports consisting of two 25 mm diameter stainless steel half-rounds that are spaced 102 $\pm$ 0.5 mm apart.

7.1.1.2 Loading Shaft - A blunt-nosed loading shaft continuous with the load cell and deflection measuring transducer which is capable of applying a preload of 30 $\pm$ 5 mN and maintaining a test load of 980 $\pm$ 50 mN within $\pm$ 5 mN using differential air pressure or other means such as electro-hydraulic for adjustment. The rise time for the test load shall be less than 0.1 s where the rise time is the time required for the load to rise from the 30 $\pm$ 5 mN preload to the 980 $\pm$ 50 mN test load. During the rise time the system shall dampen the test load to a constant $\pm$ 5 mN value.

7.1.1.3 Load Cell - A load cell with a minimum capacity of 2000 mN having a sensitivity of 1 mN mounted in-line with the loading shaft and above the fluid to measure the preload and the test load.

7.1.1.4 Linear Variable Differential Transducer (LVDT) - A linear variable differential transducer or other suitable device mounted axially above the loading shaft capable of resolving a linear movement $\leq$ 2.5 μm with a range of at least 10 mm to measure the deflection of the test beam. Digital or analog smoothing of the load and deflection data may be required to eliminate electronic noise that could otherwise affect the ability of the second order polynomial to fit the data with sufficient accuracy to provide a reliable estimate of the m-value. Averaging five or more load or deflection signals equi-spaced over a range $\pm$ 0.2 s from the reported time is acceptable to smooth the load or deflection signal.

7.1.2 Controlled-Temperature Fluid Bath - A controlled-temperature liquid bath capable of maintaining the temperature at all points within the bath between -40 and 25°C within $\pm$ 0.1°C. Placing a cold specimen in the bath may cause the bath temperature to fluctuate $\pm$ 0.2 °C from the target test temperature, consequently bath fluctuations of $\pm$ 0.2 °C during testing and iso-thermal conditioning shall be allowed.

7.1.2.1 Bath Fluid - A bath fluid that is not absorbed by or does not affect the properties of the asphalt binder tested.

7.1.2.2 Bath Agitator - A bath agitator for maintaining the required temperature homogeneity with agitation intensity such that the fluid currents do not disturb the testing process and mechanical noise caused by vibrations is less than the resolution specified in 7.1.1.4.

7.1.2.3 Circulating Bath (Optional) - A circulating bath unit separate from the test frame which pumps the bath fluid through the test bath. If used, vibrations from the circulating system shall be isolated from the bath test chamber so that mechanical noise is less than the resolution specified in section 7.1.1.4.

7.1.3 Data Acquisition System - A data acquisition system that resolves loads to the nearest 0.1 mN, beam deflection to the nearest 2.5 μm, and bath fluid temperature to the nearest 0.1°C. The system shall sense the point in time when the load is first applied (zero time) and, using this time as a reference, shall provide a record of subsequent load and deflection measurements relative to this time within $\pm$ 0.20 s. The system shall record the load and deflection at loading times of 8, 15, 30, 60, 120 and 240 s, with less than 0.1 s between load and deflection measurement times. All readings shall be an average of five or more points within $\pm$ 0.2 seconds from the loading time, e.g. for a loading time of 8 s, average 7.8, 7.9, 8.0, 8.1 and 8.2 seconds.

7.2 Temperature Measuring Equipment - A calibrated temperature transducer capable of measuring the temperature to 0.1°C over the range -40 to 25°C mounted in the immediate vicinity of the midpoint of the test specimen.

Note 1 -- Required temperature measurement can be accomplished with an appropriately calibrated platinum resistance thermometer (RTD) or a thermistor. Calibration of the RTD or thermistor can be verified as per Section 7.6. The platinum resistance thermometers meeting DIN Standard 43760 (Class A) are recommended for this purpose. The required precision and accuracy cannot be obtained unless each RTD is calibrated as a system with its respective meter or electronic circuitry.

7.3 Test Beam Molds - Test beam molds of suitable dimensions to yield demolded test beams 6.35 $\pm$ 0.05 mm thick by 12.70 $\pm$ 0.05 mm wide by 127 $\pm$ 0.5 mm long fabricated from aluminum flat stock as shown in Figure 2, or from silicone rubber as shown in Figure 3.

7.4 Stainless Steel Beams - One stainless steel beam 6.4 $\pm$ 0.1 mm thick by 12.7 $\pm$ 0.1 mm wide by 127 $\pm$ 0.5 mm long for measuring system compliance and one stainless steel beam 1.0 to 1.6 mm thick by 6.35 mm wide measured to $\pm$ 0.02 mm by 127 mm long measured to $\pm$ 0.5 mm with a known stiffness modulus, for performing periodic checks on the performance of the BBR.

7.5 Standard Masses - Four standard 50.0 or 100.0 $\pm$ 0.2-g masses for periodic BBR calibration verification.

7.6 Calibrated Thermometers - Calibrated liquid-in-glass thermometers for verification of the temperature transducer of suitable range with subdivisions of 0.1°C. These thermometers shall be calibrated in accordance with ASTM E77. ASTM thermometers 89C and 119C are suitable thermometers.

7.7 Thickness Gauge - A stepped thickness gauge for verifying the calibration of the displacement transducer as described in Figure 4.

8. Materials

8.1 Plastic Sheeting - Clear plastic sheeting 0.12 to 0.15 mm thick, for lining the interior faces of the three long aluminum mold sections. Sheeting should not be distorted by hot asphalt binder. Transparency film sold for use with laser printers has been found suitable.

8.2 Petroleum Based Grease - A petroleum based grease used to hold the plastic strips to the interior faces of the three long aluminum mold sections. **(Warning: do not use any silicone based products.)**

8.3 Glycerol-Talc Mixture - Used to coat the end pieces of aluminum molds.

8.4 Suitable bath fluids include ethanol and glycol-methanol mixtures (e.g. 60% glycol, 15% methanol, 25% water).

9. Hazards

9.1 Observe standard laboratory safety procedures when handling hot asphalt binder and preparing test specimens.

10. Preparation of Apparatus

10.1 Clean the supports, loading head and bath fluid of any particulates and coatings as necessary.

> Note 2 -- Because of the brittleness of asphalt binder at the specified test temperatures, small fragments of asphalt binder can be introduced into the bath fluid. If these fragments are present on the supports or the loading head, the measured deflection will be affected. The small fragments, because of their small size, will deform under load and add an apparent deflection to the true deflection of the beam. Filtration of the bath fluid will aid in preserving the required cleanliness.

10.2 Select the test temperature and adjust the bath fluid to the selected temperature. Wait until the temperature stabilizes and then allow the bath to equilibrate to the test temperature $\pm$ 0.2°C.

10.3 Activate the data acquisition system and load the software as explained in the manufacturer's manual for the test system.

11. Standardization

11.1 Verify calibration of the displacement transducer, load cell, and temperature transducer. Conduct each of the steps in this section each day before conducting any tests.

> Note 3 -- Calibration is usually performed by a calibration service agency. Calibration verification and quality checks may be performed by the manufacturer, other agencies providing such services, or in-house personnel using the procedures described below.

11.1.1 Verify calibration of the displacement transducer using a stepped thickness gauge of known dimensions similar to the one shown in Figure 4. Remove the loading frame from the bath. Place the gauge on a reference platform underneath the loading shaft. Measure the rise of the steps relative to the top surface of the gauge with the displacement transducer. Compare the measured values with the known dimensions of the gauge. If the difference is $\geq$ 4 μm, further calibration or maintenance is required. Report the calibration constants (μm/mV) for the displacement transducers to three significant figures. The calibration constants should be repeatable from day-to-day otherwise the operation of the system may be suspect.

11.1.2 Verify calibration of the load cell using four standard dead masses evenly distributed over the range of the load cell. Perform the verification by resting the loading shaft against the 6.35 mm thick standard steel beam and loading it with the four standard masses sequentially in four steps while recording the load after each step. If the load indicated by the data acquisition system does not agree with the force imposed by the standard masses within $\pm$ 2 mN at all steps, further calibration or maintenance is required. Report the calibration constants (mN/m) for the load transducers to three significant figures. The calibration constants should be repeatable from day-to-day otherwise the operation of the system may be suspect.

11.1.3 Verify calibration of the temperature detector by using a calibrated thermometer of suitable range meeting the requirements of Section 7.6. Immerse the thermometer in the liquid bath close to the thermal detector and compare the temperature indicated by the calibrated thermometer to the detector signal being displayed. If the temperature indicated by the thermal detector does not agree with the mercury in glass thermometer within $\pm$ 0.1°C, further calibration or maintenance is required.

11.2 Determine the compliance of the loading system by placing the 6.35 mm thick stainless steel beam on the testing supports, resting the loading tip of the loading shaft against the stainless steel beam and applying a load to the shaft of 980 $\pm$ 50 mN measured to the nearest 2 mN. The beam is relatively rigid within the range of loading applied and the range of deflection measurement specified. Therefore, any measured deflection is caused by the

load cell or other parts of the test system. Divide the deflection measured under this load by the applied load. The quotient is the compliance of the loading system in μm/mN. Use this result to calculate the deflection component that is not due to the compliance of the system. This component is automatically subtracted from the deflection measured during a test.

11.3 Perform daily a quality control check on the operation of the overall system using the 1.0 to 1.6 mm thick stainless steel beam of known modulus. Load the steel beam with 981 and 1961 mN (100 and 200 g) using the standard masses and measure the deflection at mid-span. Using the load applied and deflection measured, calculate the elastic modulus of the beam. Compare the calculated modulus to the known modulus of the beam. If the calculated modulus differs from the known modulus by 10 percent or more, the operation of the system is suspect. Perform required maintenance on the system and then repeat Section 11.

12. Preparation of Molds and Test Specimens

12.1 To prepare aluminum molds, spread a very thin layer of petroleum based grease, only sufficient to hold the plastic strips to the aluminum, on the interior faces of the three long aluminum mold sections. Place the plastic strips over the aluminum faces and rub the plastic with firm finger pressure. Assemble the mold as shown in Figure 2 using the rubber O-rings to hold the pieces of the mold together. Inspect the mold and press the plastic film against the aluminum to force out any air bubbles. If air bubbles remain disassemble the mold and recoat the aluminum faces with grease. Cover the inside faces of the two end pieces with a thin film of glycerol to prevent the asphalt binder from sticking to the aluminum end pieces. After assembly keep the mold at room temperature until pouring the asphalt binder.

12.2 To prepare silicone rubber molds, assemble the two mold sections.

12.3 If unaged binder is to be tested, obtain test samples according to T40.

12.4 Heat the material until it is sufficiently fluid to pour.

> Note 4 -- Minimum pouring temperatures that produce a consistency equivalent to that of SAE 10W30 motor oil (readily pours but not overly fluid) at room temperature are recommended. Heating unaged asphalt binders to temperatures above 135 °C should be avoided, however, with some modified asphalts or heavily aged binders pouring temperatures above 135 °C may be required. PAV residues shall be placed in TFOT pans and may be heated up to 163 °C. In all cases heating time should be minimized. These precautions will help avoid oxidative hardening and volatile loss that will further harden the sample. During the heating process the sample should be covered and stirred occasionally to ensure homogeneity.

12.5 Molding (aluminum mold) - If an aluminum mold is used, begin pouring the binder from one end of the mold and move toward the other end, slightly overfilling the mold. When pouring, hold the sample container 20 to 100 mm from the top of the mold, pour continuously toward the other end in a single pass. Allow the mold to cool 45 to 60 minutes to room temperature after pouring, and trim the exposed face of the cooled specimens flush with the top of the mold using a hot knife or a heated spatula. Discard the plastic sheeting (lining the mold sections) if they become distorted.

12.6 Molding (silicone rubber mold) - If a silicone rubber mold is used, fill the mold from the top of the mold in a slow steady manner taking care not to entrap air bubbles. Fill the mold to the top with no appreciable overfilling. Allow the mold to cool to room temperature for at least 45 minutes after pouring.

12.7 Store all test specimens in their molds at room temperature prior to testing. Schedule testing so that it is completed within 4 hours after specimens are poured.

> Note 5 -- Time-dependent increases in stiffness can occur when asphalt binders are stored at room temperature for even short periods of time. This increase in stiffness is the result of molecular associations and is referred to as steric hardening in the literature.

12.8 Just prior to testing, cool the aluminum or silicone mold containing the test specimen in a freezer or ice bath

at - 5°C ± 5°C for 5 to 10 minutes, only long enough to stiffen the asphalt binder beam so that it can be readily demolded without distortion. (Note 6) Some softer grades may require lower temperatures. Do not cool the molds containing the specimens in the test bath because it may cause temperature fluctuations in the bath to exceed ± 0.2°C.

Note 6 -- Excessive cooling may cause unwanted hardening of the beam thereby causing increased variability in the test data.

12.9 Immediately demold the specimen when it is sufficiently stiff to demold without distortion by disassembling the aluminum mold or by removing the test specimen from the silicone rubber mold.

Note 7 -- Minimize distortion of the specimen during demolding. Full contact at specimen supports is assumed in the analysis. A warped test beam yields a measured stiffness less than the actual stiffness.

13. Procedure

13.1 When testing a specimen for compliance with MP1 select the appropriate test temperature from Table 1 of MP1. After demolding, immediately place the test specimen in the testing bath and condition it at the testing temperature for 60 ± 5 minutes.

Note 8 -- Asphalt binders may harden rapidly when held at low temperatures. This effect, which is called physical hardening, is reversible when the asphalt binder is heated to room temperature or slightly above. Because of physical hardening, conditioning time must be carefully controlled if repeatable results are to be obtained.

13.2 After conditioning, place the test beam on the test supports and initiate the test. Maintain the bath at test temperature ± 0.2°C during testing.

13.3 Enter the specimen identification information, test load, test temperature, time the specimen is placed in bath test temperature, and other information as appropriate into the computer which controls the test system.

13.4 Manually apply a 30 ± 5 mN preload to the beam to ensure contact between the beam and the loading head.

Note 9 -- The specified preload on the specimen is required to ensure continuous contact between the loading shaft and the specimen. Failure to establish continuous contact within the required load range gives misleading results.

13.5 Activate the automatic test system which is programmed to proceed as follows.

13.5.1 Apply a 980 ± 50 mN initial seating load for 1 ± 0.1 second.

Note 10 -- The actual load on the beam as measured by the load cell is used in calculating the stress in the beam. The 980 ± 50 mN initial seating and test load includes the 30 ± 5 mN preload.

13.5.2 Reduce the load to 30 ± 5 mN and allow the beam to recover for 20 ± 0.1 seconds.

Note 11 -- The initial seating loads described in Sections 13.5.1 and 13.5.2 are applied and removed automatically by the computer-controlled loading system and are transparent to the operator. Data are not recorded during initial loading.

13.5.3 Apply a test load ranging from 980 ± 50 mN, and maintain the load constant to ± 5 mN for 240 seconds.

13.5.4 Remove the test load and terminate the test.

13.5.5 At the end of the initial seating, load and at the end of the test, monitor the computer screen to verify that the load on the beam in each case returns to 30 ± 5 mN.

13.6 Remove the specimen from the supports and proceed to the next test.

14. Calculation and Interpretation of Results

See Annex.

15. Report

15.1 Report data as shown in Figure A1.1 that describes individual tests including:

15.1.1 Temperature of the test bath measured 60 s after the test load is first applied, nearest 0.1°C,

15.1.2 Date and time when test load is applied,

15.1.3 File name of test data,

15.1.4 Name of operator,

15.1.5 Sample identification number,

15.1.6 Any flags issued by software during test,

15.1.7 Correlation coefficient, R^2 for log stiffness versus log time, expressed to nearest 0.000001,

15.1.8 Anecdotal comments (maximum 256 characters),

15.1.9 Report constants A, B, and C to three significant figures,

15.1.10 Difference between measured and estimated stiffness calculated as:

$$\text{(Measured-Estimated) x 100\% / Estimated} \qquad (17)$$

15.2 Report data as shown in Figure A1.1 for time intervals of 8, 15, 30, 60, 120, and 240 seconds including:

15.2.1 Time beam is placed in bath,

15.2.2 Time test started,

15.2.3 Loading time, nearest 0.1 second,

15.2.4 Load, nearest 0.1 mN,

15.2.5 Beam deflection, nearest 2 μm,

15.2.6 Measured Stiffness modulus, MPa, expressed to three significant figures,

15.2.7 Estimated Stiffness modulus, MPa, expressed to three significant figures,

15.2.8 Estimated m, nearest 0.001.

16. Precision and Bias

16.1 Precision - The research required to develop precision values in accordance with ASTM C802 has not been conducted.

16.2 Bias - The research required to establish the bias of this method has not been conducted.

17. Keywords - Flexural creep stiffness, flexural creep compliance, bending beam rheometer

Annex

1.1 A typical test result is shown in Figure A1.1. Disregard measurements obtained and the curves projected on the computer screen during the initial 8 seconds application of the test load. Data from a creep test obtained immediately after the application of the test load may not be valid because of dynamic loading effects and the finite rise time. Use only the data obtained between 8 and 240 seconds loading time for calculating S(t) and *m*.

1.2 Deflection of an elastic beam - Using the elementary bending theory, the mid-span deflection of an elastic prismatic beam of constant cross-section loaded in three-point loading can be obtained by applying equations (1) and (2) as follows:

$$\delta = PL^3/48EI \quad (1)$$

where:

δ = deflection of beam at midspan, mm
P = load applied, N
L = span length, mm
E = modulus of elasticity, MPa
I = moment of inertia, mm^4

and:

$$I = bh^3/12 \quad (2)$$

where:

I = moment of inertia of cross-section of test beam, mm^4
b = width of beam, mm
h = thickness of beam, mm

Note 12 -- The test specimen has a span to depth ratio of 16 to 1 and the contribution of shear to deflection of the beam can be neglected.

1.3 Elastic flexural modulus - According to elastic theory, calculate the flexural modulus of a prismatic beam of constant cross-section loaded at its midspan thus:

$$E = PL^3/4bh^3\delta \quad (3)$$

where:

E = time-dependent flexural creep stiffness, MPa
P = constant load, N
L = span length, mm
b = width of beam, mm
h = depth of beam, mm
δ = deflection of beam, mm

1.4 Maximum bending stress - The maximum bending stress in the beam occurs at the midspan at the top and bottom of the beam. Calculate σ thus:

$$\sigma = 3PL/2bh^2 \quad (4)$$

where:

σ = maximum bending stress in beam, MPa
P = constant load, N
L = span length, mm
b = width of beam, mm
h = depth of beam, mm

1.5 Maximum bending strain - The maximum bending strain in the beam occurs at the midspan at the top and bottom of the beam. Calculate ϵ thus:

$$\epsilon = 6\delta h/L^2 \text{ mm/mm} \quad (5)$$

where:

ϵ = maximum bending strain in beam, mm/mm
δ = deflection of beam, mm
h = thickness of beam, mm
L = span length, mm

1.6 Linear Viscoelastic Stiffness Modulus - According to the elastic-viscoelastic correspondence principle, it can be assumed that if a linear viscoelastic beam is subjected to a constant load applied at t = 0 and held constant, the stress distribution is the same as that in a linear elastic beam under the same load. Further, the strains and displacements depend on time and are derived from those of the elastic case by replacing E with 1/D(t). Since 1/D(t) is equivalent to S(t), rearranging the elastic solution results in the following relationship for the stiffness:

$$S(t) = PL^3/4bh^3\delta(t) \quad (6)$$

where:

S(t) = time-dependent flexural creep stiffness, MPa
P = constant load, N
L = Span length, mm
b = width of beam, mm
h = depth of beam, mm
$\delta(t)$ = deflection of beam, mm, and
$\delta(t)$ and S(t) indicate that the deflection and stiffness, respectively are functions of time.

1.7 Presentation of Data

1.7.1 Plot the response of the test beam to the creep loading as the logarithm of stiffness with respect to the logarithm of loading time. A typical representation of test data is shown in Figure A1.2. Over the limited testing time from 8 to 240 seconds, the plotted data shown in Figure A1.2 can be represented by a second order polynomial as follows:

$$\log S(t) = A + B[\log(t)] + C[\log(t)]^2 \quad (7)$$

and, the slope, *m*, of the logarithm of stiffness versus logarithm time curve is equal to (absolute value):

$$|m(t)| = d[\log S(t)]/d[\log(t)] = B + 2C[\log(t)] \quad (8)$$

where:

S(t) = time-dependent flexural creep stiffness, MPa
t = time in seconds
A, B, and C = regression coefficients

Smoothing the data may be required to obtain smooth curves for the regression analysis as required to determine an *m* value. This can be done by averaging 5 readings taken at the reported time $\pm$0.1 and $\pm$0.2 seconds.

1.7.2 Obtain the constants A, B, and C from the least squares fit of equation 7. Use data equally spaced with respect to the logarithm of time to determine the regression coefficients in equations 7 and 8. Determine experimentally the stiffness values used for the regression to derive the coefficients A, B, and C and to, in turn, calculate values of *m* after loading times of 8, 15, 30, 60, 120, and 240 seconds.

1.8 Calculation of Regression Coefficients, estimated stiffness values and *m*.

1.8.1 Calculate the regression coefficients A, B, and C in equations 7 and 8 and the denominator D as follows:

$$A = [S_y(S_{x2}S_{x4} - S_{x3}^2) - S_{xy}(S_{x1}S_{x4} - S_{x2}S_{x3}) + S_{xxy}(S_{x1}S_{x3} - S_{x2}^2)] / D \quad (9)$$

$$B = [6(S_{xy}S_{x4} - S_{xxy}S_{x3}) - S_{x1}(S_yS_{x4} - S_{xxy}S_{x2}) + S_{x2}(S_yS_{x3} - S_{xy}S_{x2})] / D \quad (10)$$

$$C = [6(S_{x2}S_{xxy} - S_{x3}S_{xy}) - S_{x1}(S_{x1}S_{xxy} - S_{x3}S_y) + S_{x2}(S_{x1}S_{xy} - S_{x2}S_y)] / D \quad (11)$$

$$D = 6(S_{x2}S_{x4} - S_{x3}^2) - S_{x1}(S_{x1}S_{x4} - S_{x2}S_{x3}) + S_{x2}(S_{x1}S_{x3} - S_{x2}^2) \quad (12)$$

where, for loading times of 8, 15, 30, 60, 120, and 240 seconds:

S_{x1} = log 8 + log 15 + ... log 240
S_{x2} = $(\log 8)^2 + (\log 15)^2 + \ldots (\log 240)^2$
S_{x3} = $(\log 8)^3 + (\log 15)^3 + \ldots (\log 240)^3$
S_{x4} = $(\log 8)^4 + (\log 15)^4 + \ldots (\log 240)^4$
S_y = log S(8) + log S(15) + ... log S (240)
S_{xy} = log S(8)(log (8)) + log S(15) log (15) + ... log S(240) log (240)
S_{xxy} = $[\log (8)]^2$ log S(8) + $[\log (15)]^2$ log S(15) + ... $[\log (240)]^2$ log S(240)

1.8.2 Calculate the estimated stiffness at 8, 15, 30, 60, 120, and 240 seconds as:

$$\log S(t) = A + B[\log(t)] + C[\log(t)]^2 \quad (13)$$

1.8.3 Calculate the estimated *m* value at 8, 15, 30, 60, 120, and 240 seconds as the absolute value of

$$m_1 = B + 2C[\log(t)] \quad (14)$$

1.8.4 Calculate the fraction of the variation in the stiffness explained by the quadratic model as:

$$R^2 = 1.00 - \left[\frac{[\log S(8) - \log S(8)]^2 + \ldots [\log S(240) - \log S(240)]^2}{[\log S(8) - \log(\bar{S})]^2 + \ldots [\log S(240) - \log(\bar{S})]^2} \right] \quad (15)$$

1.8.5 Calculate $\bar{S}$ the average of the stiffness values at 8, 15, 30, 60, 120, and 240 seconds as:

$$\log \bar{S} = [\log S(8) + \ldots \log S(240)]/6 \quad (16)$$

1.8.6 Use the estimated values of the stiffness and *m* at 60 seconds for specification purposes. Measured and estimated stiffness values should agree to within 2 percent. Otherwise the test is considered suspect.

TEST INFORMATION

Project : testing	Target Temp: 23.0°C	Conf. Test : 2.199e+008
Operator : jsy	Actual Temp: 14.8°C	Date : 09/17/93
Specimen: plastic beam b	Soak Time : 0.0 sec	Load Const: 0.24
Time : 11:47:03	Beam Width : 12.70 mm	Defl Const: 0.0024
Date : 09/18/93	Thickness : 6.35 mm	Date : 09/17/93
File : 0818934.DAT		

RESULTS

t Time (sec)	P Force (N)	d Defl (mm)	Measured Stiffness (kPa)	Estimated Stiffness (kPa)	Difference (%)	*m*-value
8	.9859	.9126	87030.0	87060.0	.03532	0.176
15	.9894	1.022	77990.0	77930.0	-.08120	0.175
30	.9913	1.158	68960.0	68990.0	.04809	0.175
60	.9910	1.308	61110.0	61110.0	.004487	0.174
120	.9908	1.475	54150.0	54150.0	-.001551	0.174
240	.9906	1.664	48010.0	48000.0	-.005077	0.174

Regression Coefficients:
a = 5.100 b = -.1784 c = .001020 R^2 = 0.999996
- CANNON BENDING BEAM RHEOMETER - P to print - ESC to continue

Figure A1.1: Typical Test Report

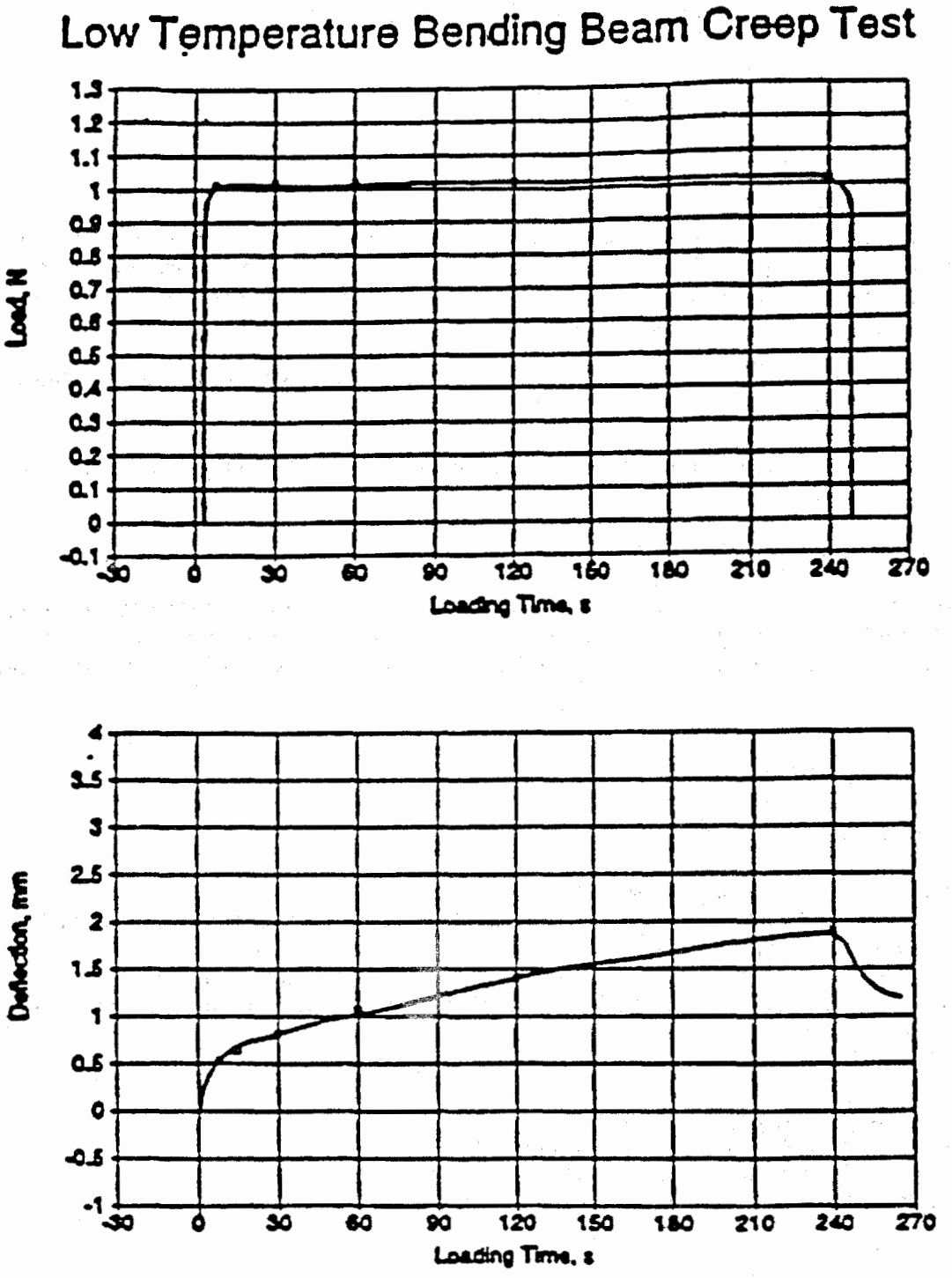

Figure A1.2: Typical Load and Deflection Plots

Standard Test Method for
Determining the Fracture Properties of
Asphalt Binder in Direct Tension (DT)

AASHTO Designation: TP3[1]

1. Scope

1.1 This test method covers the determination of the failure strain and failure stress of asphalt binders by means of a direct tension test. It is applicable to material having a failure strain less than ten percent and can be used with unaged material or with material aged using T240 (RTFOT), T179 (TFOT), or PP1 (PAV). The test apparatus is designed for testing within the temperature range from -40°C to 25°C.

1.2 This test method is limited to asphalt binders containing particulate material having dimensions less than 250 μm.

1.3 This test method is not valid for specimens exhibiting a strain to failure outside the defined brittle-ductile range ($\geq$ 10 percent).

1.4 The values stated in SI units are to be regarded as the standard.

1.5 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. Reference Documents

2.1 AASHTO Standards:

MP1 Specification for Performance-Graded Asphalt Binder
PP1 Practice for Accelerated Oxidative Aging of Asphalt Binder Using a Pressure Aging Vessel (PAV)
T40 Practice for Sampling Bituminous Materials
T179 Test Method for Effect of Heat and Air on Asphalt Materials (Thin-Film Oven Test)
T240 Test Method for Effect of Heat and Air on Rolling Film of Asphalt (Rolling Thin-Film Oven Test)

2.2 ASTM Standards:

E1 Specification for ASTM Thermometers
E4 Practice for Load Verification of Testing Machines
E83 Method of Verification and Classification of Extensometers
E220 Method for Calibration of Thermocouples by Comparison Techniques

2.3 DIN Standards

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[1] *This standard is based on SHRP Product 1005.*

43760

2.4 MIL Standards

45662

45662A

xxx standard for plastics

3. Terminology

3.1 Definitions

3.1.1 asphalt binder, n — an asphalt-based cement that is produced from petroleum residue either with or without the addition of non-particulate organic modifiers.

3.2 Description of Terms Specific to this Standard

3.2.1 brittle, adj — refers to type of failure in a direct tension test where the stress-strain curve is essentially linear up to the point of failure and the failure is by sudden rupture of the test specimen without appreciable reduction in cross-section of the specimen.

3.2.2 brittle-ductile, adj — refers to type of failure in a direct tension test where the stress-strain curve is curvilinear and the failure is by sudden rupture of the test specimen. Limited reduction in cross-section of the specimen occurs before rupture.

3.2.3 ductile, adj — refers to a type of failure in a direct tension test where the specimen does not rupture but fails by flow at large strains.

3.2.4 engineering strain, n — refers to the axial strain resulting from the application of a tensile load and calculated as the change in length caused by the application of the tensile load divided by the original unloaded length of the specimen without any correction for reduction in cross-section

3.2.5 failure, n — refers to the point at which the tensile load reaches a maximum value as the test specimen is pulled at a constant rate of elongation

3.2.6 failure stress, n — the tensile stress on the test specimen when the load reaches a maximum value during the test method specified in this standard

3.2.7 failure strain, n — the tensile strain corresponding to the failure stress

4. Summary of Test Method

4.1 This method describes the procedure used to measure the strain at failure and stress at failure in an asphalt binder test specimen pulled at a constant rate of elongation. Test specimens are prepared by pouring hot asphalt binder into a suitable mold. Two plastic inserts are used to grip the asphalt binder during the test and to transfer the tensile load from the test machine to the test specimen.

4.2 This test procedure was developed for asphalt binders at temperatures where they exhibit brittle or brittle-ductile failure. A brittle or brittle-ductile failure will result in a fracture of the test specimen as opposed to a ductile failure in which the specimen simply stretches without fracturing. The test is not applicable at temperatures where failure is by ductile flow.

4.3 A non-contact extensometer is used to measure the elongation of the test specimen as it is pulled in tension at

a constant rate of elongation of 1 mm/min. The maximum load developed during the test is monitored and the tensile strain and stress in the test specimen when the load reaches a maximum is reported as the failure strain and failure stress respectively.

5. Significance and Use

5.1 The strain at failure is a measure of the amount of elongation that an asphalt binder can sustain without cracking. Strain at failure is used as a criterion for specifying the low temperature properties of asphalt binder in accordance with MP1.

5.2 The test is designed to identify the temperature region where the asphalt binder has limited ability to elongate without cracking. In the asphalt binder specification a lower limit is placed on the allowable strain to failure at a specified temperature and rate of elongation.

5.3 For evaluating an asphalt binder for conformance to MP1, the elongation rate is 1.0 mm/min and the test temperature is selected from Table 1 of MP1 according to the grade of asphalt binder. Other rates of elongation and test temperatures may be used to test asphalt binders.

6. Apparatus

6.1 Direct Tension Test System - A direct tension test system consisting of (1) a displacement-controlled tensile loading machine, (2) a specimen gripping system, (3) a chamber for environmental conditioning and testing, and (4) load measuring and recording devices, (5) elongation measuring and recording devices, (6) a temperature detection device, and (7) data acquisition and display devices.

6.1.1 Tensile Loading Machine - A tensile loading machine with a controlled-displacement loading frame capable of producing at least a 500 N load is required. The platen movement shall be controlled with either one or two motor-driven screws or with a hydraulic-controlled actuator. Screw-type loading frames are typically used for this test although a hydraulic closed-loop testing frame is acceptable if the requirements specified in this standard are met. The loading frame shall produce a cross-head speed of 1.00 $\pm$ 0.05 mm/min. Additional crosshead speeds may be supplied but are not required. The testing frame shall be equipped with an automatic return feature that returns the cross-head to a reference position such that the center to center spacing of the loading pins is 100.0 mm $\pm$ 0.1 mm. The testing frame shall be equipped with two standards with sufficient clear space between the standards so that an environmentally controlled chamber with dimensions given in Section 6.1.3 can be placed between the standards.

6.1.2 Specimen Gripping System - The gripping system shall produce a self-aligning uniaxial test load, accept the plastic end units described in Section 6.1.2.1 and be designed so that test specimens can be easily mounted in the machine. The system shall include two grips. Each grip shall include a specially-shaped pin that is mounted rigidly to the upper and lower platens of the testing machine. Figure 1 shows the grip and loading pin assembly and specifies the dimensions of the top and bottom grips. One grip shall be fixed and remain stationary during the test while the other grip is displaced at the desired rate.

6.1.2.1 Specimen Plastic End Inserts: PMMA (**Plexiglas™**) end inserts having the dimensions specified in Figure 2 shall be used on both ends of the test specimen to transfer the tensile load to the asphalt binder. Each end insert contains a precisely machined hole and slot. The diameter of the hole is slightly larger than the diameter of the pin. The slots on the top and bottom inserts allow two spots of laser light to pass the test specimen and shine upon the receiver. Gripping of the specimen is accomplished through the bond (adhesion) between the asphalt binder test specimen and the plastic end insert. Each end insert mounts on a specially shaped pin that is part of the gripping system. The specimen is attached to the top and bottom grips by positioning the end inserts in the test machine such that the plastic inserts fit onto the pins and are indexed against the face of the grips. Matching the coefficient of thermal expansion of the asphalt binder and the inserts is necessary to reduce thermal shrinkage stresses at the interface that otherwise cause bond failures.

Note 1 -- PMMA (**Plexiglas™**), meeting MIL STD-5545, has been found acceptable for making the inserts.

It is readily available, opaque to the laser beam, has a satisfactory coefficient of thermal expansion, and is easy to machine.

6.1.3 Environmental Conditioning and Testing Chamber - The environmental conditioning and testing chamber shall be capable of controlling temperature and humidity and isothermal conditioning test specimens prior to testing. It should be on the order of 300 mm wide by 200 mm deep by 450 mm high and shall completely enclose the specimen and gripping system. It shall be capable of controlling test temperatures between -40°C and ambient with variations within the chamber $\leq$ 0.2°C. It shall be equipped with a cooling system that has the capacity to reduce the chamber temperature from ambient to -40°C within 40 minutes and to change the chamber temperature from -30 to -40°C or from -40 to -30°C within 10 minutes. Mechanical cooling or liquid nitrogen may be used to cool the chamber. It shall have a dehumidifying system with a capacity such that the formation of frost on the interior of the chamber, the test specimen, or any of the test fixtures is eliminated.

6.1.3.1 The chamber shall be capable of storing a minimum of 16 test specimens on a rack which is thermally isolated from the walls and floors of the chamber such that heat conducted from the walls and floors of the chamber does not affect the temperature of the stored specimens.

Note 2 -- A demolded test specimen is placed on Plexiglas™, Teflon or other plastic plate (with approximate dimensions of 6 mm thick by 20 mm wide by 100 mm long) and transferred to the environmentally controlled test chamber for thermal conditioning. The use of a transfer plate will minimize deformation during handling. Each plate, when the above dimensions are used, accommodates up to four demolded test specimens.

6.1.3.2 The chamber shall be fitted with a front-opening door for maintenance and standardization purposes and an access port that allows for insertion of the operator's hand and forearm to position test specimens on the storage shelf for conditioning and to position test specimens on the grips for testing. The access port shall be designed so that changes in chamber temperature are $\leq$ 0.2°C during an operation in which the operator's hand or forearm is inserted into or removed from the chamber. Visual access to the interior of the test chamber shall be provided to permit proper mounting of test specimens and test monitoring. The test specimen elongation is measured with an optical laser. Use of the laser requires optical glass windows on two sides of the temperature chamber so that a beam of laser light can be passed through the chamber without distorting the beam.

6.1.4 Load Measuring and Recording Devices - Load shall be measured with a load cell having a minimum capacity of 500 N and a sensitivity of 0.1 N. The load cell shall be calibrated at least annually in accordance with ASTM E4. The load and elongation shall be monitored with the data acquisition system such that they can be resolved to 1 percent of the failure load and elongation, respectively. Once the test has started, the data acquisition system shall be able to detect the point in time when the load starts to change as a result of elongation in the sample. This shall be accomplished by monitoring the load cell signal with time. A change in the load signal equivalent to 1 to 2 N (threshold load) shall be used to mark the point in time where zero reading of the extensometer is obtained. The point in time where the peak load is obtained will be captured by the data acquisition system and the accumulated elongation from the zero reading to the elongation corresponding to the peak load shall be used to calculate the failure strain. Once the test is complete the device shall display the strain at failure. Peak load typically range from 10 to 100 N depending on the test temperature, grade, aging, and source of the binder. Stress and strain shall be displayed to the nearest 0.1 N.

6.1.5 Elongation Measuring and Recording Devices - Specimen elongation shall be measured with a laser light transmitter and receiver. The non-contact laser extensometer shall be calibrated at least annually according to ASTM E83, MIL - STD 45662 and MIL - STD 45662A. The transmitter shall produce a vertical plane of laser light which is monitored by the receiver and shall be arranged so that the test specimen interrupts the plane of laser light except for the slots in the insert. Thus, two spots of laser light are transmitted to the receiver. The receiver shall constantly monitor the relative position of the two spots of laser light to produce a voltage proportional to the distance between the two nearest edges of the spots of light. This voltage shall be converted to elongation by a controller attached to the receiver using a calibration factor determined by the manufacturer and verified when the extensometer is calibrated. The laser measurement range shall be 30 to 60 mm with an accuracy $\leq$ 0.005 mm. Elongations shall be recorded to the nearest 0.005 mm.

6.1.6 Temperature Detection Device - The temperature detection device shall be a calibrated resistance thermal

detector (RTD) readable and accurate to 0.1°C. (Note 3) The RTD shall be mounted inside the environmental chamber in the immediate vicinity of the test specimen. Cabling to the RTD shall be of sufficient length that the bulb of a total immersion mercury-in-glass thermometer can be held adjacent to the RTD for standardization purposes (See Section 9.1.3).

Note 3 -- Required temperature measurement can be accomplished with an appropriately calibrated platinum resistance thermometer (RTD) or thermistor. Platinum resistance thermometers meeting DIN Standard 43760 (Class A) are recommended for this purpose. The required precision and accuracy cannot be obtained unless each RTD or thermistor is calibrated as a system with its respective meter or electronic circuitry (See Section 9.1.3).

6.1.7 Data Acquisition and Display Device - The data acquisition and display device shall display the load and elongation selected by the operator (stress and strain) on an LED (or other appropriate computer controlled display) during the time the test specimen is loaded. It shall detect the peak load and capture the elongation associated with the peak load. The maximum skew time between the load and corresponding elongation shall be 0.015 s.

6.1.7.1 If the data acquisition component consists of an IBM-compatible computer it shall have three A/D channels; one for load (the load cell), one for elongation (the laser), and one for temperature (the RTD). Data shall be stored in ASCII format.

6.1.7.2 Display of Stress-Strain Curve - The data acquisition and display system shall be capable of displaying a stress-strain curve in units of stress (MPa) versus strain (percent). This may be accomplished using the video screen of the data acquisition computer or with an x-y recorder. If a recorder is used the units may be in volts but in this case the test file shall contain the calibration factor in MPa/volt and percent strain/volt for both the x and y axes.

6.2 Specimen Molds - The specimen molds shall be manufactured from silicone rubber available from Dow Corning (HSII RTV Moldmaking Rubber 20:1 Kit, white in color). Silicone rubber is used because it is dimensionally stable, provides an excellent non-stick surface for asphalt binder and is easy to clean. Specimens and molds shall have the dimensions specified in Figure 3.

6.3 Specimen Mold Support Plates - Aluminum plates 5 to 10 mm thick to support the rubber molds when molding test specimens are required.

6.4 Specimen Storage Plates - Plexiglas™, Teflon or other plastic plates for transferring and storing test specimens in the environmental chamber.

6.5 Calibrated thermometer - A calibrated liquid-in-glass thermometer of suitable range with subdivisions of 0.1°C is required for verification of the temperature transducer. This thermometer shall be calibrated in accordance with ASTM E77. An ASTM thermometer 62C thermometer is suitable.

6.6 Load Verification Equipment - Known dead masses (traceable to NIST) ranging 10 to 500 $\pm$ 0.1 N (1 to 50 $\pm$ 0.1 kg) are required for verifying the calibration of the load cell.

7. Materials

7.1 Solvent (Varsol™ or mineral spirits) or a degreasing spray cleaner formulated for use on plastic for cleaning molds, plastic end inserts and aluminum plates

7.2 Cleaning cloths (cotton) for wiping molds, end inserts and plates

8. Hazards - Use standard laboratory safety procedures required for handling the hot asphalt binder when preparing test specimens and required safety procedures when cleaning with solvents or degreasers.

9. Calibration and Standardization

9.1 Verify calibration of the extensiometer, load cell, and temperature transducer.

Note 4 -- Calibration is usually performed by a calibration service agency. Calibration verification, system standardization, and quality checks may be performed by the manufacturer, other agencies providing such services, or in-house personnel using the procedures described below.

9.1.1 Load cell - Verify calibration of the load cell at least every 6 months using dead masses suspended from the load cell.

9.1.2 Non-Contact Extensometer - Verify calibration of the non-contact extensometer at least every month using the fixture shown in Figure 4. Place the fixture in the test machine and apply a 20 to 50 N load on the fixture to hold the fixture firmly in the grips. Insert the gage in the fixture and measure the short dimension of the gage with the laser. Remove the gage from the fixture and measure the long dimension. Check the length of the gage as measured with a similar reference measurement made on the gage during the last calibration. If the verification measurement and the reference measurement differ by more than 0.006 mm, either calibration or maintenance is required.

Note 5 -- Measurements should be made at -12 °C. Allow sufficient time for gage to achieve thermal equilibrium.

9.1.3 Temperature Detector - Verify calibration of the temperature detector at least every six months by comparing the output of the RTD with a calibrated mercury in glass thermometer in accordance with ASTM E220. Place the thermometer in the environmentally controlled chamber and hold the RTD in intimate contact with the bulb of the thermometer with a rubber O-ring or other suitable technique. When the thermometer and the temperature detection device have reached equilibrium, compare the temperature indicated on the readout for the detector to the temperature observed by reading the thermometer through the observation port on the door of the chamber. If the temperature indicated by the thermal detector does not agree with the mercury in glass thermometer within ± 0.1°C, further calibration or maintenance is required.

9.1.4 Verify the speed of the crosshead using a dial gauge. Mount a dial gauge on a fixed portion of the testing machine in a manner such that the stem of the dial gauge senses the movement of the crosshead. Be careful to ensure that the movement of the stem of the dial gauge is parallel to the movement of the crosshead and that the dial gauge is firmly attached to the fixed portion of the testing machine. Mounting the dial gauge on a magnetic base is a convenient method for attaching the dial gauge to the testing machine. Select the desired crosshead speed and start operating the machine at the desired speed. Using a stopwatch, as the machine is running, start the stopwatch and simultaneously obtain an initial reading of the dial gauge. Approximately one minute later obtain a second reading and simultaneously stop the stopwatch. Calculate the speed of the crosshead by dividing the difference between the initial and final dial gauge reading in mm by the stopwatch time in minutes.

10. Preparation of Samples and Test Specimens

10.1 Preparing Test Samples - If unaged binder is to be tested, obtain test samples according to T40.

10.1.2 Anneal the asphalt binder from which the test specimen is obtained by heating until sufficiently fluid to pour. Annealing prior to testing removes reversible molecular associations (steric hardening) that occur during normal storage at ambient temperature.

Note 6 -- Minimum pouring temperatures that produces a consistency equivalent to that of SAE 10W30 motor oil (readily pours but not overly fluid) at room temperature are recommended. The specific temperature will depend on the grade of binder and its prior aging history if any. Temperatures less that 135°C are desirable, however, temperatures above 135°C may be required for some modified asphalt binders or heavily aged binders.

10.2 Place the plastic end inserts into both ends of the mold. Place the molds and inserts in to a 163 °C oven for

30 minutes.

10.3 After heating, place the mold on a 6 to 7-mm thick aluminum plate. Do not preheat the aluminum plate. Pour hot asphalt binder into the mold starting from one end of the mold and moving across the mold in a single pass. Pour the specimen in a continuous stream to avoid entraining air bubbles or gaps. Complete the pouring operation as quickly as possible to avoid any excessive drop in the temperature of the asphalt binder. Stop pouring when the asphalt is slightly above the top surface of the mold.

10.4 After pouring the test specimen allow the entire assembly to cool on the benchtop at ambient temperature for approximately one hour. Do not quench the specimen to achieve ambient temperature.

10.5 As soon as the specimen has cooled to room temperature, trim off the excess asphalt binder with a straight edge (e.g. a flat cheese cutter or spatula) so that the asphalt binder is flush with the top of the mold. Use care during the trimming operation so that the asphalt binder is not pulled away from the mold and that the bond between the plastic inserts and the asphalt binder is not damaged. Trim off the specimen in a consistent manner. Pull the cheese cutter along the long axis of the sample flush with the surface of the mold to remove the excess asphalt binder. After trimming remove all debris or extraneous asphalt binder from the holes or slots in the plastic insert.

Note 7 -- Caution: Excessive downward pressure during trimming will distort the sample mold.

10.6 Immediately prior to demolding, place the mold containing the specimen in a freezer at -5 $\pm$ 5°C until the specimen is sufficiently stiff to demold without distorting the sample. Some softer grades may require lower temperatures. Time in the freezer should not exceed 15 minutes. After demolding place the sample on the flat specimen storage plate (see Section 6.4). Measurement of specimen dimensions after demolding is not necessary since dimensional tolerances are closely controlled in the molding process.

11. Procedure

11.1 Set the environmental chamber at the desired testing temperature and wait until it stabilizes to within $\pm$ 0.2°C of the desired test temperature. When testing for compliance with MP1, use the test temperatures specified in Table 1 of MP1.

11.2 Immediately after demolding, place the test specimens in the chamber on the plastic storage plate and condition the specimens at the test temperature for 1 hour $\pm$ 10 minutes. Adhere carefully to time schedule to avoid testing variability that is caused by physical hardening.

11.3 After 1 hour $\pm$ 10 min, mount the specimen on the pins using the environmental chamber hand access port so that the back face of the insert is centered on the mounting pin. Do not open the chamber door to handle or mount the specimen because that will produce excessive temperature fluctuations which will take time to stabilize and lead to variable thermal histories. Handle the specimens with rubber surgeons gloves to protect the operator's fingers and to minimize heating of the specimen. Handle the specimen by touching only the plastic inserts--do not touch the asphalt binder.

Note 8 -- Air currents from the circulation fan may cause the inserts to move after being placed on the mounting pins. A silicone rubber or foam washer that remains flexible at the test temperature will help hold the insert against the face of the grip. It is important that the insert be centered on the pin (flush against the face of the grip) in order for the load to be applied axially through the center of the test specimen. A suitable washer may be cut from silicone rubber or foam sheeting with a cork borer. The washer may be 5 - 10 mm thick with an outer diameter of approximately 10 mm. The inside diameter of the washer shall be sufficient to provide a friction fit on the 5-mm portion of the pin. The washer should slide easily on the pin providing only sufficient force to hold the insert in place during the test.

11.4 Select the desired deformation rate and load the specimen to failure. Select a deformation rate of 1.00 $\pm$ 0.05 mm/min when testing for compliance with MP1. If a test specimen fails outside the gage area of the specimen (from throat to throat), discard the test.

11.5 Alternate loading procedure - Apply a preload to the test specimen by mounting the specimen as previously described and applying an elongation sufficient to develop a 10 to 20 N load; this may reduce testing variability. As soon as the 10 to 20 N load is reached stop the movement of the platen and allow the load to relax until it is no longer detectable. The time required to relax the load will depend on the stiffness of the test specimen. Once the load has relaxed continue the test as described in section 11.4.

11.6 The strain at failure is easily identified as the strain at peak load (maximum stress) when the failure is by fracture (i.e. breaks into two pieces on failure). However, when the specimen does not fracture but reaches a maximum stress and then flows without fracture the strain at failure is recorded as the strain corresponding to the maximum stress. In some cases, the maximum stress may occur at strains greater than 10 percent. In this case do not continue the test beyond 10 percent strain and simply record the failure stress as "greater than 10 percent". If the asphalt binder can be stretched to 10 percent without fracture, it meets the requirements of MP1 at the test temperature.

11.7 **If the plastic specimen end inserts are to be reused,** After testing break off the asphalt binder from the inserts while the asphalt binder is still cold. Discard the asphalt binder and clean the end inserts by soaking them in solvent and wiping with a soft cloth. (Do not use acetone, TCE, or toluene, since such solvents will dissolve plastic inserts.) After wiping the inserts, use a detergent soap solution to remove any oil film residue left by the mineral spirit cleaner. Alternatively, use a degreasing spray cleaner formulated for use on plastic. Clean the plastic inserts thoroughly. Grease-film on the asphalt bonding area can create a weak bond causing bond failures.

12. Calculation

12.1 Compute the failure stress by dividing the failure load by the original area of the test specimen cross-section as shown in equation 1:

$$\sigma_f = P_f/A \qquad (1)$$

where:

σ_f = failure stress, MPa
P_f = failure load, N
A = original area of cross-section, mm^2

Note 9 -- For specimens used in this test A = 3.6 mm^2

12.2 Compute the failure strain by dividing the elongation at failure by the original gage length, as shown in equation 2:

$$\epsilon_f = \delta_f/L \qquad (2)$$

where:

ϵ_f = failure strain, mm/mm
δ_f = elongation at failure, mm
L = gage length, mm

Note 10 -- For specimens used in this test, the effective gauge length, L, is assumed to be 27.0 mm. This is an effective gauge length that represents the portion of the specimen that contributes to the majority of the strain. Strain values determined in this test may vary slightly from the actual strain at the point of fracture because of the assumed value for the gauge length.

13. Report

13.1 Report the following information:

13.1.1 the sample identification,

13.1.2 the date and time of test,

13.1.3 the test temperature, nearest 0.1°C,

13.1.4 the average rate of elongation, nearest 0.01 mm/min.,

13.1.5 the average failure strain, nearest 0.01 percent,

Note 11 -- Direct tension testing of multiple specimens may produce obvious outliers. Until a statistically valid procedure is developed for considering outliers, discard any strain at failure values that are obvious outliers.

13.1.6 the average failure stress, nearest 0.01 MPa,

13.1.7 the average peak load, nearest N, and

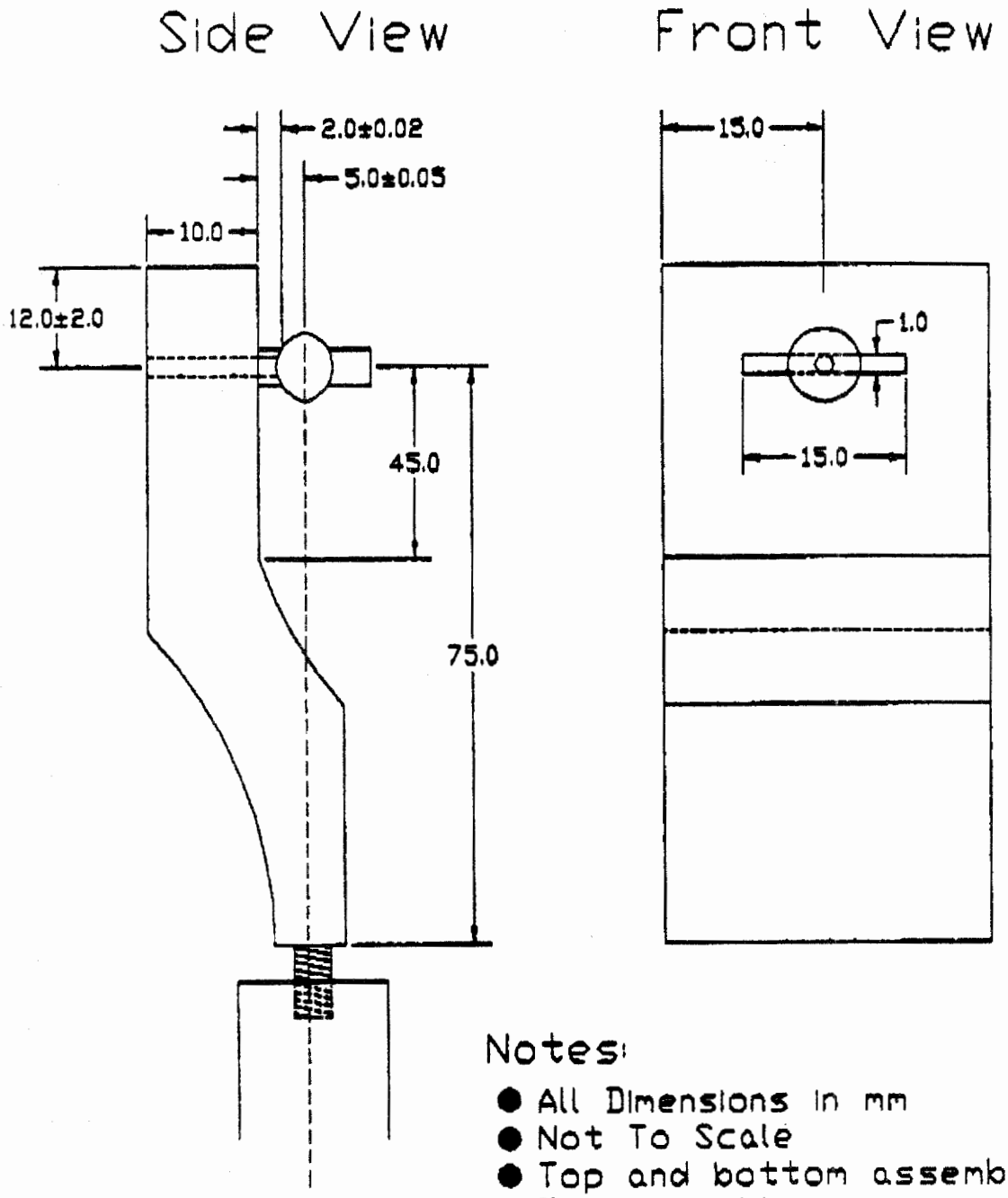
13.1.8 the type of fracture observed (fracture or no fracture).

14. Precision and Bias

14.1 Precision - The research required to develop precision estimates for this test method has not been conducted.

14.2 Bias - The research required to establish the bias of this method has not been conducted.

15. **Keywords** - direct tension, fracture, failure, asphalt binder, thermal cracking, failure strain



Notes:

- All Dimensions in mm
- Not To Scale
- Top and bottom assemblies are identical. Two assemblies are required.
- Unless otherwise indicated, assume a tolerance of ±0.05 mm

Figure 1a: Top and Bottom Grip and Loading Pin Assembly

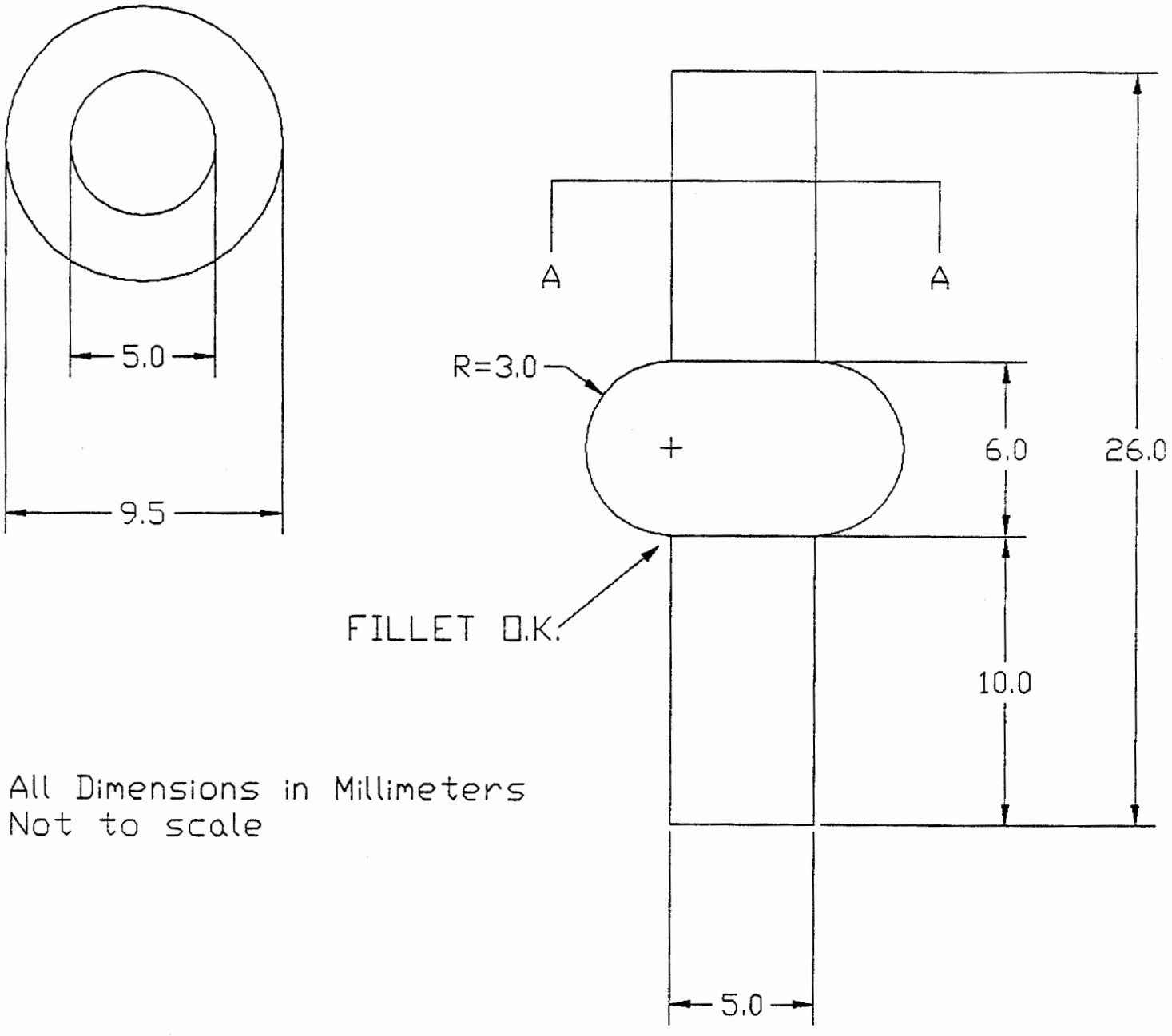
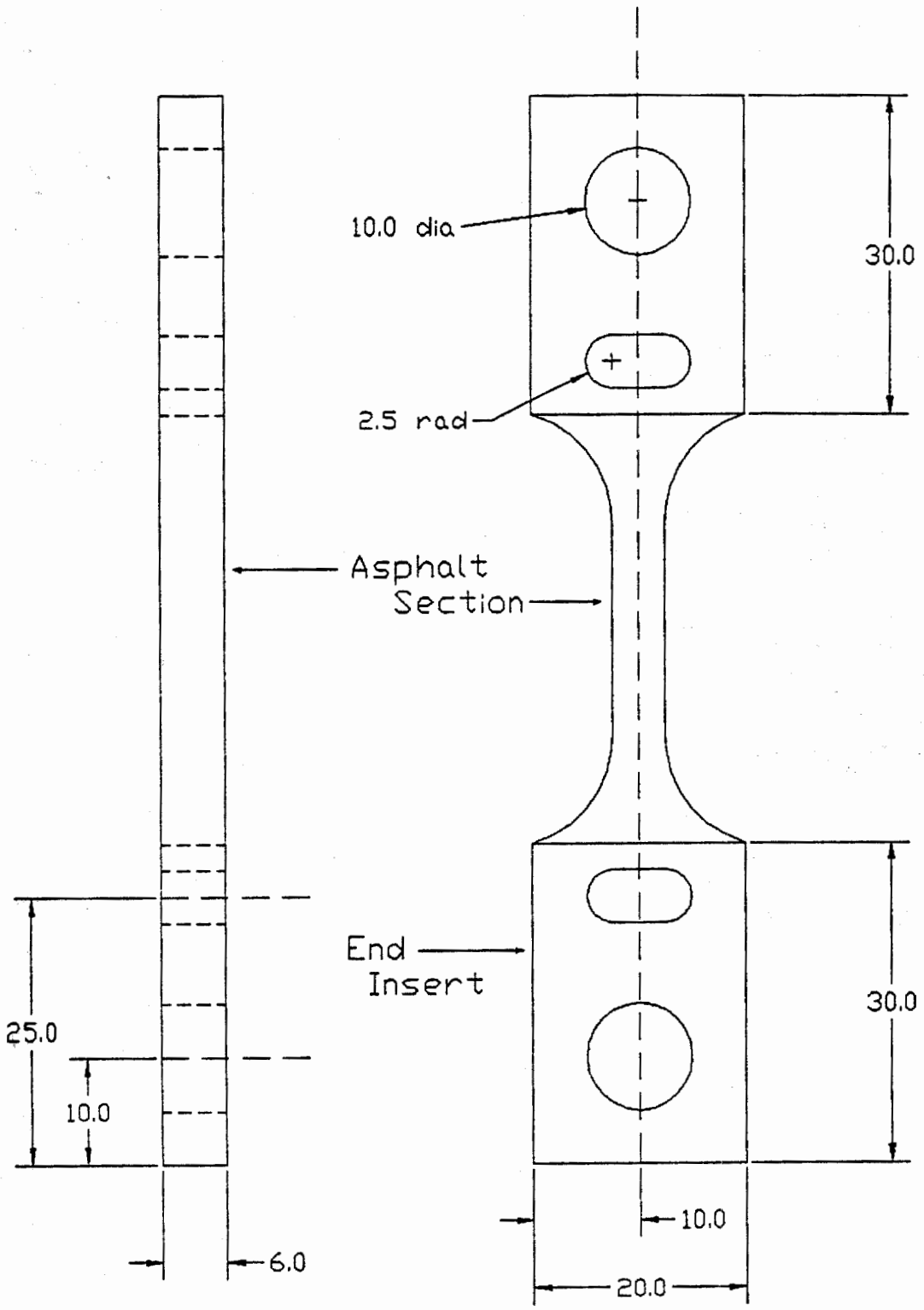


Figure 1b Specially Designed Pins Used to Mount the Specification Type Direct Tension Specimen.



Notes:

- All Dimensions in mm
- Not to Scale
- Unless otherwise indicated, assume a tolerance of ± 0.05

Figure 2: Plastic End Inserts

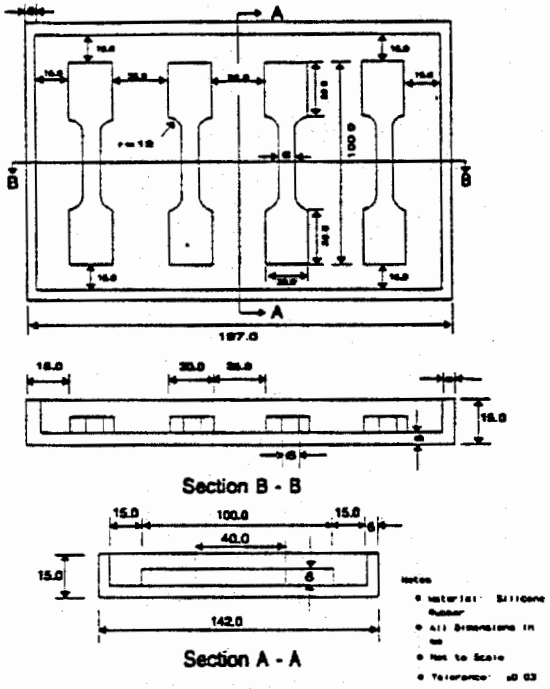


Figure 3: Direct Tension Specimen Mold

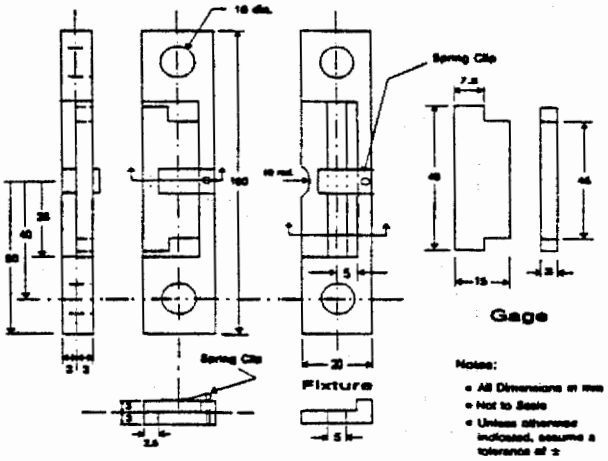
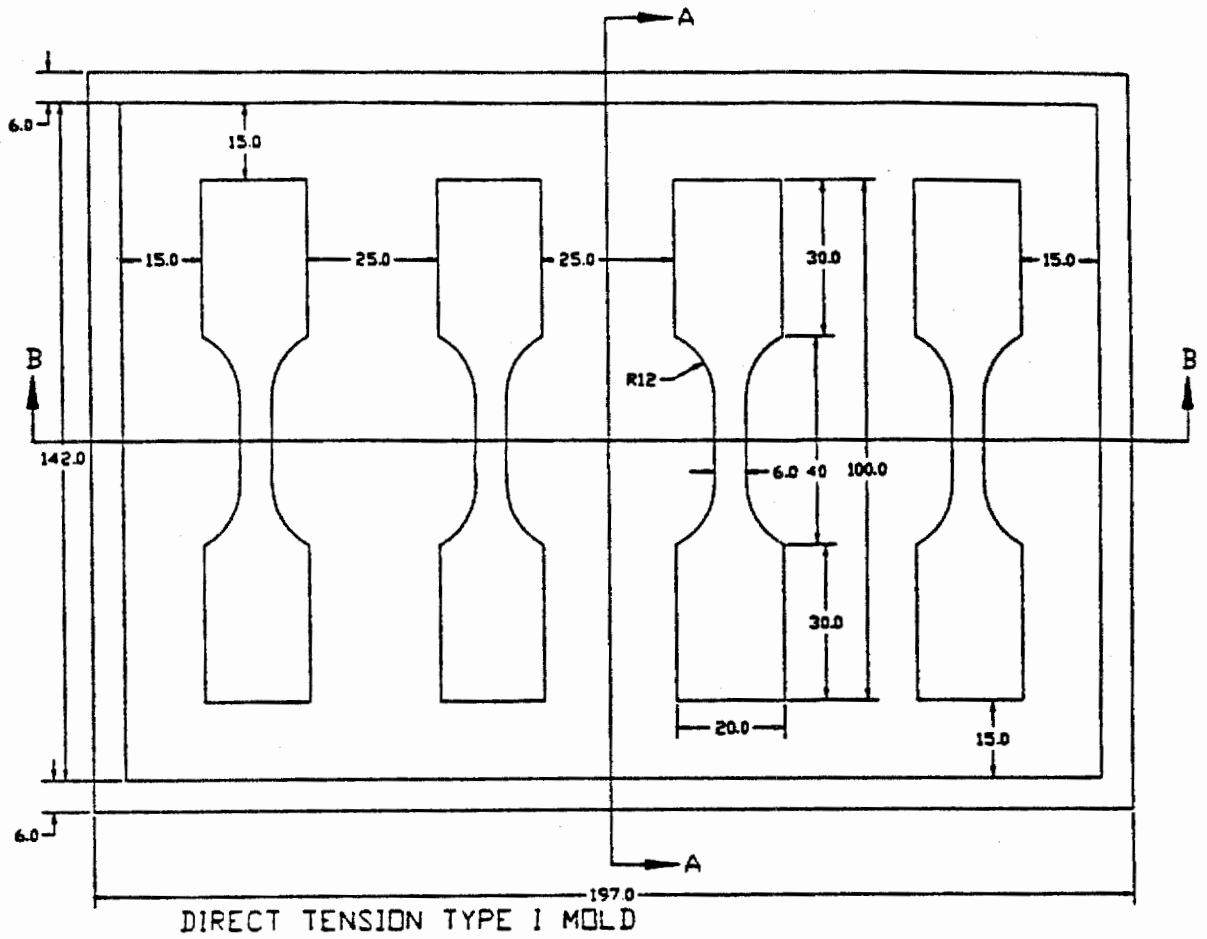
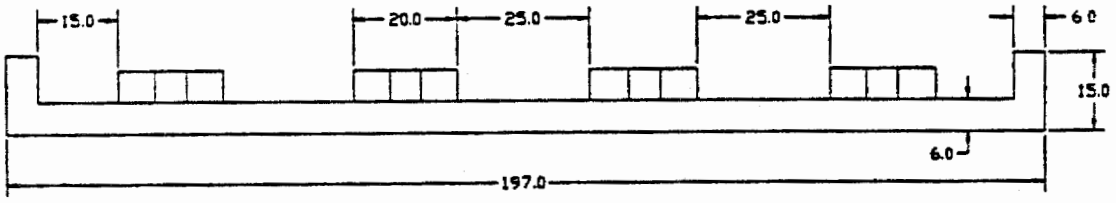
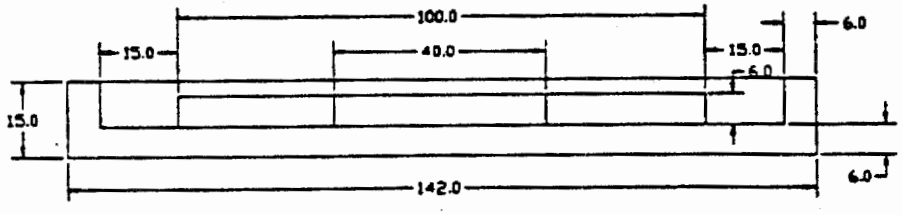


Figure 4: Extensometer Verification Device





SECTION B-B



SECTION A-A

BI-CO No.: 1992-12
MATERIAL: ALUMINIUM
ALL DIMENSIONS IN mm
TOLERENCE: ±0.03mm

Figure 5. Illustration of specimen mold dimensions.

Standard Test Method for
Determining the Rheological Properties
of Asphalt Binder Using a
Dynamic Shear Rheometer (DSR)

AASHTO Designation: TP5[1]

1. Scope

1.1 This test method covers the determination of the dynamic shear modulus and phase angle of asphalt binder when tested in dynamic (oscillatory) shear using parallel plate test geometry. It is applicable to asphalt binders having dynamic shear modulus values in the range from 100 Pa to 10 MPa. This range in modulus is typically obtained between 5°C and 85°C. This test method is intended for determining the linear viscoelastic properties of asphalt binders as required for specification testing and is not intended as a comprehensive procedure for the full characterization of the viscoelastic properties of asphalt binder.

1.2 This standard is appropriate for unaged material or material aged in accordance with T240, T179, or PP1.

1.3 Particulate material in the asphalt binder is limited to particles with longest dimensions less than 250 μm.

1.4 The values stated in SI units are to be regarded as the standard.

***1.5** This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. Reference Documents

2.1 AASHTO Standards

MP1	Specification for Performance-Graded Asphalt Binder
T40	Practice for Sampling Bituminous Materials
T179	Test Method for Effect of Heat and Air on Asphalt Materials (Thin-Film Oven Test)
T240	Test Method for Effect of Heat and Air on Rolling Film of Asphalt (Rolling Thin-Film Oven Test)
TP1	Test Method for Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer
PP1	Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)
PP6	Practice for Grading or Verifying the Performance Grade of an Asphalt Binder

2.2 ASTM Standards

E1	Specification for ASTM Thermometers
E220	Method for Calibration of Thermocouples by Comparison Techniques

2.3 DIN Standards

43760

3. Terminology

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[1] *This standard is based on SHRP Product 1007.*

3.1 Definitions

3.1.1 asphalt binder, n — an asphalt-based cement that is produced from petroleum residue either with or without the addition of non-particulate organic modifiers.

3.2 Descriptions of Terms Specific to this Standard

3.2.1 complex shear modulus, G^* — ratio calculated by dividing the absolute value of the peak-to-peak shear stress, τ, by the absolute value of the peak-to-peak shear strain, γ.

3.2.2 phase angle, δ — the angle in radians or degrees, between a sinusoidally applied strain and the resultant sinusoidal stress in a controlled-strain testing mode, or between the applied stress and the resultant strain in a controlled-stress testing mode.

3.2.3 loss shear modulus, G'' — the complex shear modulus multiplied by the sine of the phase angle expressed in degrees. It represents the component of the complex modulus that is a measure of the energy lost (dissipated during a loading cycle).

3.2.4 storage shear modulus, G', — the complex shear modulus multiplied by the cosine of the phase angle expressed in degrees. It represents the in-phase component of the complex modulus that is a measure of the energy stored during a loading cycle.

3.2.5 parallel plate geometry, n — refers to a testing geometry in which the test sample is sandwiched between two relatively rigid parallel plates and subjected to oscillatory shear

3.2.6 oscillatory shear, n — refers to a type of loading in which a shear stress or shear strain is applied to a test sample in an oscillatory manner such that the shear stress or strain varies in amplitude about zero in a sinusoidal manner.

3.2.7 linear viscoelastic, adj — within the context of this specification refers to a region of behavior in which the dynamic shear modulus is independent of shear stress or strain

3.2.8 molecular association, n — refers to associations that occur between asphalt binder molecules during storage at ambient temperature. Often referred to as steric hardening in the asphalt literature, molecular associations can increase the dynamic shear modulus of asphalt binders. The extent of molecular association is asphalt specific and may be apparent even after a few hours of storage.

4. Summary of Test Method

4.1 This standard contains the procedure used to measure the complex shear modulus (G^*) and phase angle (δ) of asphalt binders using a dynamic shear rheometer and parallel plate test geometry.

4.2 The standard is suitable for use when the dynamic shear modulus varies between 100 Pa and 10 MPa. This range in modulus is typically obtained between 5°C and 85°C, dependent upon the grade, test temperature, and conditioning (aging) of the asphalt binder.

4.3 Test specimens 1 mm thick by 25 mm in diameter or 2 mm thick by 8 mm in diameter are formed between parallel metal plates. During testing, one of the parallel plates is oscillated with respect to the other at pre-selected frequencies and rotational deformation amplitudes (or torque amplitudes). The required amplitude depends upon the value of the complex shear modulus of the asphalt binder being tested. The required amplitudes have been selected to ensure that the measurements are within the region of linear behavior.

4.4 The test specimen is maintained at the test temperature to within $\pm$ 0.1°C by positive heating and cooling of the upper and lower plates.

4.5 Oscillatory loading frequencies using this standard can range from 1 to 100 rad/s using a sinusoidal waveform. Specification testing is performed at a test frequency of 10 rad/s. The complex modulus (G^*) and phase angle (δ) are calculated automatically as part of the operation of the rheometer using proprietary computer software supplied by the equipment manufacturer.

5. Significance and Use

5.1 The test temperature for this test is related to the temperature experienced by the pavement in the geographical area for which the asphalt binder is intended.

5.2 The complex shear modulus is an indicator of the stiffness or resistance of asphalt binder to deformation under load. The complex shear modulus and the phase angle define the resistance to shear deformation of the asphalt binder in the linear viscoelastic region. Other linear viscoelastic properties, such as the storage modulus (G'), or the loss modulus (G"), can be calculated from the complex modulus and the phase angle. The loss modulus (G") is a measure of the energy dissipated during each loading cycle.

5.3 The complex modulus and the phase angle are used to calculate performance-related criteria in accordance with MP1.

6. Apparatus

6.1 Dynamic Shear Rheometer (DSR) Test System - A dynamic shear rheometer test system consisting of parallel metal plates, an environmental chamber, a loading device, and a control and data acquisition system.

6.1.1 Test plates - Metal test plates with smooth polished surfaces. One 8.00 $\pm$ 0.05 mm in diameter and one 25.00 $\pm$ 0.05 mm in diameter. The base plate in some rheometers is a flat plate. A raised portion 2 to 5 mm high with the same radius as the upper plate is recommended. The raised portion makes it easier to trim the specimen and may improve test repeatability.

6.1.2 Environmental Chamber - A chamber for controlling the test specimen temperature, by heating (in steps or ramps), or cooling (in steps or ramps), to maintain a constant specimen environment. The medium for heating and cooling the specimen in the environmental chamber is a gas or liquid (Note 1) that will not affect asphalt binder properties. The temperature in the chamber may be controlled by the circulation of fluid or conditioned gas; nitrogen or water is acceptable. When air is used, a suitable drier must be included to prevent condensation of moisture on the plates and fixtures and, if operating below freezing, the formation of ice. The environmental chamber and the temperature controller shall control the temperature of the specimen, including thermal gradients within the sample, to an accuracy of $\pm$ 0.1°C. The chamber shall completely enclose the top and the bottom plates to minimize thermal gradients.

Note 1 -- A circulating bath unit separate from the DSR which pumps the bath fluid through the test chamber may be required if a fluid medium is used.

6.1.2.1 Temperature Controller - A temperature controller capable of maintaining specimen temperatures within 0.1°C for test temperatures ranging from 5 to 85°C.

6.1.2.2 Temperature Detector - A resistance thermal detector (RTD) mounted inside the environmental chamber, in intimate contact with the fixed plate, with a range of 5 to 85°C, readable and accurate to the nearest 0.1°C. This detector shall be used to control the temperature in the chamber and provide a continuous readout of temperature during the mounting, conditioning, and testing of the specimen.

Note 2 - Platinum RTDs meeting DIN Standard 43760 (Class A) or equal, are recommended for this purpose. The RTD shall be calibrated as an integral unit with its respective meter or electronic circuitry.

6.1.2.3 Reference Thermal Detector - A thermistor, RTD, or thermocouple as described in sections 9.1.1.2.1,

9.1.1.2.2, or 9.1.1.2.3 shall be used.

6.1.3 Loading device - The loading device shall apply a sinusoidal oscillatory load to the specimen at a frequency of 10.0 $\pm$ 0.1 rad/s. If frequencies other than 10 rad/s are used, the frequency shall be accurate to 1 percent. The loading device shall be capable of providing either a stress controlled or strain controlled load. If the load is strain controlled, the loading device shall apply a cyclic torque sufficient to cause an angular rotational strain accurate to within 100 μrad of the strain specified. If the load is stress controlled, the loading device shall apply a cyclic torque accurate to within 10 mN•m of the torque specified. Total system compliance at 100 N•m torque shall be $<$ 2 mrad/N•m.

6.1.4 Control and Data Acquisition System - The control and data acquisition system shall provide a record of temperature, frequency, deflection angle and torque. Devices used to measure these quantities shall meet the accuracy requirements specified in Table 1. In addition, the system shall calculate and record the shear stress, shear strain, complex shear modulus (G^*) and phase angle (δ). The system shall measure and record G^*, in the range of 100 Pa to 10 MPa, to an accuracy of 0.5 percent or less and the phase angle, in the range of 0 to 90 degrees, to an accuracy of 0.1 degree.

Table 1 - Control & Data Acquisition System Requirements

Quantity	Accuracy
Temperature	0.1°C
Frequency	1 percent
Torque	10 mN•m
Deflection angle	100 μrad

6.2 Specimen Mold (Optional) - A silicone rubber mold for forming asphalt binder test specimens having a diameter approximately equal to the diameter of the upper test plate and a height approximately equal to 1.5 times the width of the test gap.

6.3 Specimen Trimmer - A specimen trimmer with a straight edge at least 4 mm wide.

6.4 Calibrated Temperature Detector - A calibrated thermocouple, thermistor, or RTD with a thickness or diameter $\leq$ 2.0 mm is suitable for measuring the temperature of a dummy specimen or sample of asphalt binder. Thermocouples and thermistors are not reliable to $\pm$ 0.1°C unless calibrated to a standard traceable to the National Institute of Standards and Technology (NIST) and must be calibrated with associated meters or electronic circuitry. Platinum RTD's are typically not suitable because they are too large to fit in the gap between the test plates in the DSR.

7. Hazards

7.1 Standard laboratory caution should be used in handling the hot asphalt binder when preparing test specimens.

8. Preparation of Apparatus

8.1 Prepare the apparatus for testing in accordance with the manufacturer's recommendations. Specific requirements will vary for different DSR models and manufacturers.

8.2 Mount the test plates on the test fixtures and tighten firmly.

8.3 Select the testing temperature according to the grade of the asphalt binder or according to the preselected testing schedule. (Note 3) Allow the DSR to reach a stabilized temperature within $\pm$ 0.1°C of test temperature.

Note 3 -- Specification MP1 and Practice PP6 provide guidance on the selection of test temperatures.

8.4 With the test plates at the test temperature or the middle of the expected testing range, establish the zero gap level (1) by manually spinning the moveable plate, and while the moveable plate is spinning, close the gap until the removable plate touches the fixed plate (The zero gap is reached when the plate stops spinning completely.), or, (2) for rheometers with normal force transducers, by closing the gap and observing the normal force and after establishing contact between the plates, setting the zero gap at approximately zero normal force.

8.5 Move the plates apart and establish a gap setting of 1 mm plus 0.05 mm (for 25-mm diameter test specimens) or 2 mm plus 0.05 mm (for 8-mm diameter test specimens).

Note 4 -- The frame, detectors, and fixtures in the DSR change dimension with temperature causing the zero gap to change with changes in temperature. Adjustments in gap are not necessary when measurements are made over a limited range of temperatures. The gap should be set at the test temperature or, when tests are to be conducted over a range of temperatures, the gap should be set at the middle of the expected range of test temperatures. For most instruments no gap adjustment is needed as long as the test temperature is within $\pm$ 12°C of the temperature at which the gap is set.

9. Calibration and Standardization

9.1 Perform the following calibration and verification procedures at least every six months:

9.1.1 Temperature - Prepare a dummy specimen of asphalt binder or use silicone wafer following standard procedures. Use the dummy specimen only for temperature verification measurements. (Dynamic shear measurements are not valid if a temperature detector is inserted into the asphalt binder.) Verify the specimen temperature indicated by the DSR RTD in trial runs using a calibrated temperature detector inserted into the dummy specimen.

9.1.1.1 Compare temperature measurements obtained from the dummy specimen and the DSR RTD. Using the temperature measured inside the dummy specimen as the reference temperature, apply an appropriate temperature correction to the temperature measurement indicated by the DSR RTD if they do not agree within $\pm$ 0.1°C.

9.1.1.2 Thermal gradients within the rheometer and the difficulty of calibrating the instrument RTD while it is mounted in the rheometer (see Note 5) require a direct measurement of the temperature between the plates using a dummy specimen and a reference thermal detector. This is accomplished by placing a dummy specimen between the plates and reading the temperature in the dummy specimen with a reference thermal detector. A thermistor, RTD, or thermocouple as described in sections 9.1.1.2.1, 9.1.1.2.2, or 9.1.1.2.3 shall be used as the reference thermal detector. Adjust the temperature in the chamber to the minimum temperature that will be used for testing and allow the chamber to come to thermal equilibrium. Read the instrument RTD and the temperature of the dummy specimen. Increase the temperature in increments of no more than 6°C and repeat the measurements to cover the range of test temperatures. Using the resulting measurements, obtain the temperature offset between the instrument RTD and the reference thermal detector inserted between the plates. This offset will not be a constant but will vary with test temperature. Offset the thermal controller on the rheometer so that the target test temperature is obtained between the plates.

Note 5 -- The RTD and its meter can be calibrated by a commercial vendor. Verification of calibration can be obtained by comparing the output from the RTD with a calibrated ASTM mercury in glass thermometer in accordance with ASTM E220. A stirred water bath is suitable for calibrating the thermal detector. Select a partial immersion mercury-in-glass thermometer with an appropriate range (ASTM 90C; 0 to 30°C or ASTM 91C; 20 to 50°C) and place the thermal detector and the thermometer in the stirred water bath. Fasten the detector to the glass thermometer with a rubber band or rubber O-ring. Allow the bath, detector, and thermometer to come to thermal equilibrium and record the temperature of the glass thermometer and the readout from the thermal detector. The temperature in the bath shall be constant to within 0.1 degree.

9.1.1.2.1 A silicone wafer 2 mm thick by 25 mm in diameter containing a thermistor calibrated to the nearest 0.1°C shall be inserted between the plates as the dummy specimen. Use a thin coating of petroleum jelly to ensure good

thermal contact. A suitable thermistor mounted in a silicone wafer is available from Cannon Instruments as Part Number 9728-V95.

9.1.1.2.2 A wafer-shaped RTD shall be mounted between the plates and used as described in section 6.1.2.3.1. The RTD must be calibrated as described in Note 5 to the nearest 0.1°C. A suitable RTD is available from Omega as part Number RTD FN105. This RTD is not waterproof and must be dipped in hot asphalt prior to calibration. To obtain measurements, the RTD is mounted in the rheometer within the asphalt binder sample. After mounting the sample and trimming excess asphalt binder proceed with the temperature measurements as described in section 6.1.2.3.

9.1.1.2.3 A thermocouple probe shall be used to measure the sample temperature by inserting the probe into a sample mounted in the rheometer as described in section 10. The thermocouple must be calibrated at 3 month intervals using the procedure described in Note 5 to the nearest 0.1°C. When obtaining the sample temperature the cabling and instrumentation must remain unchanged from that used during the calibration. To make a sample temperature reading insert the thermocouple in the asphalt binder between the plates and proceed as described in Section 6.1.2.3. A suitable thermocouple probe is available from Omega as part Number HYP1-30-1/2-T-G-60-SMP-M.

9.1.2 Calibrate the load transducer in accordance with the directions and fixtures supplied with the apparatus.

9.1.3 Calibrate the strain transducer in accordance with the directions and fixtures supplied with the apparatus.

9.1.4 Verify the overall calibration of the DSR using suitable reference fluids with viscoelastic properties similar to asphalt binder. Do not attempt to verify individual load or deflection detectors with a reference fluid. Suitable standards have not been identified.

> Note 6 -- Reference fluids exhibiting moduli and phase angles within the range of measurement may be used for verification purposes. Because reference fluids do not have the same temperature dependency as asphalt binder, caution must be used in interpreting the results obtained from such fluids.

10. Preparing Samples and Test Specimens

10.1 Preparing Test Samples - If unaged binder is to be tested, obtain test samples according to T40.

10.1.1 Anneal the asphalt binder from which the test specimen is obtained by heating until sufficiently fluid to pour the required specimens. Annealing prior to testing removes reversible molecular associations (steric hardening) that occur during normal storage at ambient temperature. Do not exceed a temperature of 163°C. Cover the sample and stir it occasionally during the heating process to ensure homogeneity and to remove air bubbles. Minimize the heating temperature and time to avoid hardening the sample.

> Note 7 -- Minimum pouring temperatures that produce a consistency equivalent to that of SAE 10W30 motor oil (readily pours but not overly fluid) at room temperature are recommended. Heating unaged asphalt to temperatures above 135°C should be avoided, however, with some modified asphalts or heavily aged binders, pouring temperatures above 135°C may be required.

10.1.2 Cold material from storage containers must be annealed prior to usage. Structure developed during storage can result in overestimating the modulus by as much as 50 percent.

10.2 Preparing Test Specimens -- Carefully clean and dry the surfaces of the test plates so that the specimen adheres to both plates uniformly and strongly. Bring the chamber to approximately 45°C so that the plates are preheated prior to the mounting of the test specimen. This will provide sufficient heat so that the asphalt binder may be squeezed between the plates for trimming and to ensure that the asphalt binder adheres to the plates. Serrated plates or otherwise roughened plates are not necessary to ensure load transfer between the asphalt binder and the plates as long as the plates are clean and dry when the test specimen is prepared. Prepare a test specimen using one of the methods specified in Sections 10.2.1 or 10.2.2.

10.2.1 Remove the removable plate and, while holding the sample container approximately 15 mm above the test plate surface, pour the asphalt binder at the center of the upper test plate continuously until it covers the entire plate except for an approximate 2 mm wide strip at the perimeter. (Note 8) Wait several minutes for the specimen to stiffen and then mount the test plate in the rheometer for testing.

Note 8 -- An eye dropper or syringe may be used to transfer the hot asphalt binder to the plate.

10.2.2 Pour the hot asphalt binder into a silicone rubber mold that will form a pellet having a diameter approximately equal to the diameter of the upper test plate and a height approximately equal to 1.5 times the width of the test gap. Allow the silicone rubber mold to cool to room temperature. Remove the specimen from the mold and center the pellet on the lower plate of the DSR.

Note 9 -- The filled mold may be chilled in a freezer to facilitate demolding of softer grades of asphalt binder. Chill the mold in the freezer only the minimum time needed to facilitate demolding the specimen.

10.3 Test Specimen Trimming - After the specimen has been placed on one of the test plates as described above, move the test plates together to squeeze the asphalt mass between the two plates. Move the plates together until the gap between plates equals the testing gap plus 0.05 mm.

10.3.1 Trim the specimen by moving a heated trimming tool around the upper and lower plate perimeters while trimming excess asphalt. The tool may be heated on a hot plate or with a flame.

Note 10 -- The calculated modulus is proportional to the fourth power of the specimen radius. Carefully trim the specimen to insure that the measurements are reliable.

10.3.2 When the trimming is completed, decrease the gap by 0.05 mm to the desired testing gap. This will cause a slight bulging of the asphalt binder at the periphery of the test specimen.

11. Procedure

11.1 Bring the specimen to the test temperature $\pm$ 0.1°C. See Note 4.

Note 11 -- The gap should be set at the starting test temperature (Section 11.1.1) or at the middle of the expected range of test temperatures (Section 11.1.2). See Note 5 for guidance on setting the gap. Typically, reliable test results may be obtained with a single sample, in an 8-mm or 25-mm plate, using temperatures within 12°C of the temperature at which the gap is set.

11.1.1 When testing a binder for compliance with MPI, select the appropriate test temperature from Table 1 of MP1.

11.1.2 When conducting a temperature sweep, start at a mid-range test temperature and increase or decrease the test temperature to cover the desired range of test temperatures. (See Sections 6 and 7 in PP6.)

11.2 Set the temperature controller to the desired test temperature, including any offset as required by Section 9.1.1.2. Allow the temperature indicated by the RTD to come to the desired temperature. The test shall be started only after the temperature has remained at the desired temperature $\pm$ 0.1 °C for at least 10 minutes. After temperature equilibration anneal the specimen for 5 minutes.

Note 12 -- It is impossible to specify a single equilibration time that is valid for DSR's produced by different manufacturers. The design (fluid bath or air oven) of the environmental control system and the starting temperature will dictate the time required to reach the test temperature.

11.3 Strain Control Mode - When operating in a strain controlled mode, determine the strain value according to the value of the complex modulus. Control the strain within 20 percent of the target value calculated by equation 1.

$$\gamma, \text{ percent} = 12.0/(G^*)^{0.29} \quad (1)$$

where:

γ = shear strain in percent
G* = complex modulus in kPa

11.3.1 When testing specimens for compliance with MP1, select an appropriate strain value from Table 2. Software is available with the dynamic shear rheometers that will control the strain automatically without control by the operator.

Table 2 - Target Strain Values

Material	G*, kPa	Strain, percent	
		Target Value	Range
Original Binder	1.0	12	9 to 15
RTFO Residue	2.2	10	8 to 12
PAV Residue	5.0	1	0.8 to 1.2

11.4 Stress Control Mode - When operating in a stress controlled mode, determine the stress level according to the value of the complex modulus. Control the stress within 20 percent of the target value calculated by equation 2.

$$\tau = 0.12(G^*)^{0.71} \quad (2)$$

where:

τ = shear stress in kPa
G* = complex modulus in kPa

11.4.1 When testing specimens for compliance with MP1 select an appropriate stress level from Table 3. Software is available with the dynamic shear rheometers that will control the stress level automatically without control by the operator.

Table 3 - Target Stress Levels

Material	G*, kPa	Stress, kPa	
		Target Level	Range
Original Binder	1.0	0.12	0.09 to 0.15
RTFO Residue	2.2	0.22	0.18 to 0.26
PAV Residue	5.0	50.	40. to 60.

11.5 When the temperature has equilibrated, condition the specimen by applying the required strain for 10 cycles at a frequency of 10 rad/s. (Note 13) Obtain a test measurement by recording data for an additional 10 cycles. Reduce the data obtained for the second 10 cycles to produce a value for the complex modulus and phase angle. Typically a Fast Fourier Transform (FFT) is used to reduce the data. Multiple measurements may be obtained to verify that the sample is properly prepared. Debonding between the plates and the binder or fracture in the sample can result in a decrease in the modulus with repeat measurements. Some asphalt binders may exhibit a reduced modulus with continued application of shear stresses (multiple measurements). The data acquisition system automatically acquires and reduces the data when properly activated. When conducting tests at more than one frequency, start testing at the lowest frequency and increase to the highest frequency.

Note 13 -- The standard frequency of 10 rad/s is used when testing binder for compliance with MP1.

11.6 The data acquisition system specified in 7.3.7 automatically calculates G^* and δ from test data acquired when properly activated.

11.7 Initiate the testing immediately after preparing and trimming the specimen. The testing at subsequent temperatures should be done as quickly as possible to minimize the effect of molecular associations (steric hardening) that can cause an increase in modulus if the specimen is held in the rheometer for a prolonged period of time. When testing at multiple temperatures all testing should be completed within four hours.

12. Interpretation of Results

12.1 The dynamic modulus and phase angle depend upon the magnitude of the shear strain; the modulus and phase angle for both unmodified and modified asphalt cement decrease with increasing shear strain as shown in Figure 1. A plot such as that shown in Figure 1 can be generated by gradually increasing the load or strain amplitude, thereby producing a strain sweep. It is not necessary to generate such sweeps during normal specification testing, however, such plots are useful for verifying the limits of the linear region.

12.2 A linear region may be defined at small strains where the modulus is relatively independent of shear strain. This region will vary with the magnitude of the complex modulus. The linear region is defined as the range in strains where the complex modulus is 95 percent or more of the zero-strain value.

12.3 The shear stress varies linearly from zero at the center of the plates to a maximum at the extremities of the plate perimeter. The shear stress is calculated from the applied or measured torque, measured or applied strain, and the geometry of the test specimen.

13. Report

13.1 Provide a complete identification and description of the material tested including name code, source and type of sample container.

13.2 Describe the instrument used for the test including model number, whether it is a constant strain or constant stress rheometer, the type of environmental chamber, and other information needed to describe the rheometer.

13.3 The strain and stress levels specified in Tables 2 and 3 have been selected to ensure a common reference point that has been shown to be within the linear region for plain and modified asphalt binders. Some systems may not be linear within this region. When this situation is observed report the modulus at the recommended stress or strain levels but report that the test conditions were outside the linear region.

13.4 For each test, report the following:

13.4.1 the test plate diameter, nearest 0.1 mm and test gap, nearest 1μm,

13.4.2 the test temperature, nearest 0.1°C,

13.4.3 the test frequency, nearest 0.1 rad/s,

14.4.4 the strain amplitude, nearest 0.01 percent, or torque, nearest mN•m,

13.4.5 the complex modulus (G^*) for the ten measurements, kPa to three significant figures, and

13.4.6 the phase angle (δ) for the second ten cycles, nearest 0.1 degrees.

14. Precision and Bias

14.1 Precision - The research required to develop estimates of precision has not been conducted.

14.2 Bias - The research required to establish the bias of this method has not been conducted.

15. Keywords - dynamic shear rheometer, DSR, complex modulus, asphalt binder

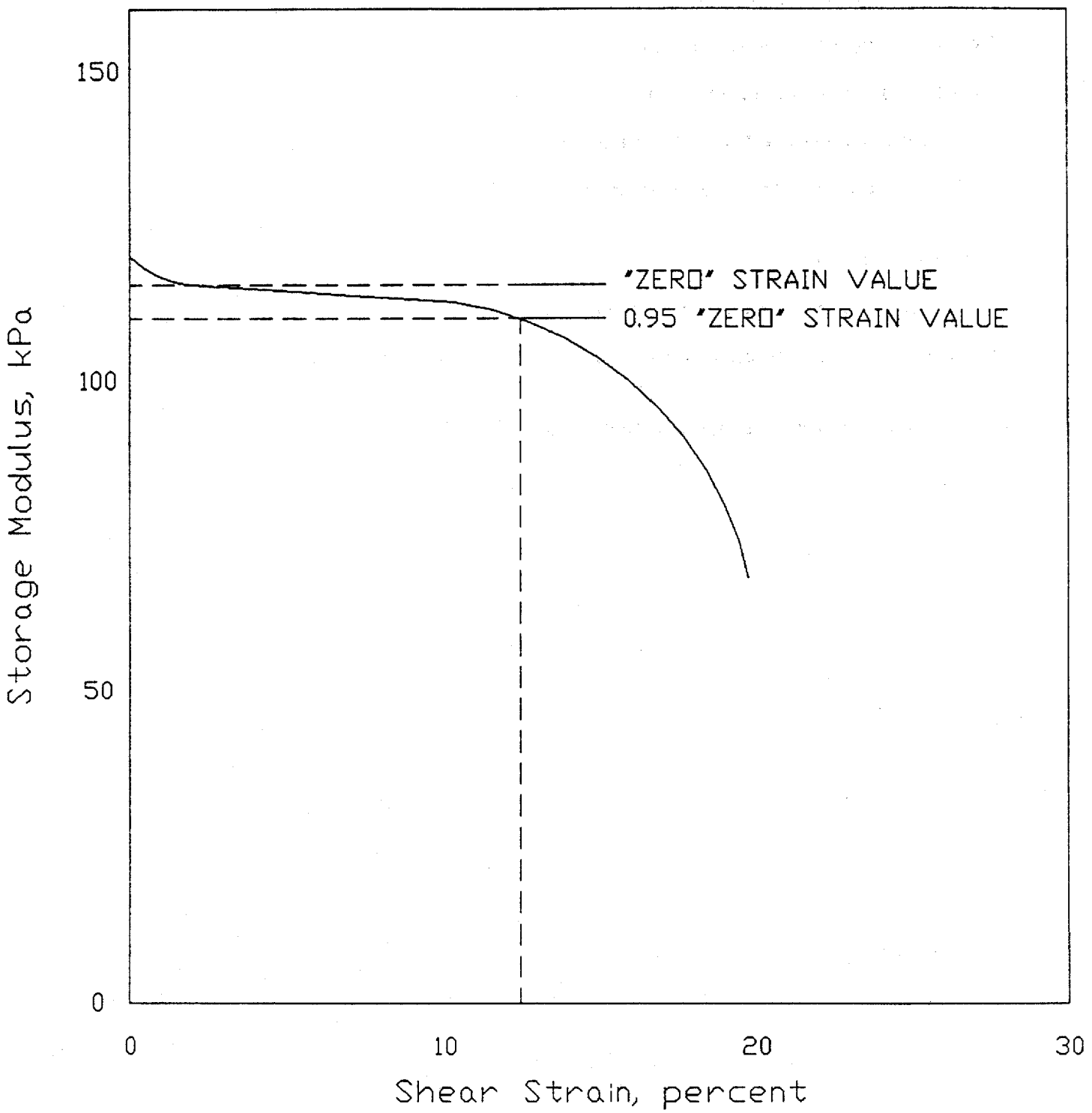

Figure 1: Example of a Strain Sweep Test Used to Define the Linear Visco-elastic Limit for Dynamic Mechanical Testing of Asphalt

Standard Specification for
Performance Graded Asphalt Binder

AASHTO Designation: MP1[1]

1. Scope - This specification covers asphalt binders graded by performance. Grading designations are related to average 7-day maximum pavement design temperatures and minimum pavement design temperatures.

Note 1 -- For asphalt cements graded by penetration at 25°C, see M20. For asphalt cements graded by viscosity at 60°C see M226.

Note 2 -- Guide PP5 provides information on the evaluation of modified asphalt binders.

Note 3 -- Guide PP6 provides information for determining the performance grade of an asphalt binder.

2. Reference Documents

2.1 AASHTO Documents:

PP5	Guide for the Laboratory Evaluation of Modified Asphalt Systems
PP6	Guide for Grading or Verifying the Performance Grade of an Asphalt Binder
PPX	Selection of Asphalt Binders (Being Developed)
M20	Specification for Penetration Graded Asphalt Cement
M226	Specification for Viscosity Graded Asphalt Cement
PP1	Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)
T40	Practice for Sampling Bituminous Materials
T44	Solubility of Bituminous Materials in Organic Solvents
T48	Method for Flash and Fire Points by Cleveland Open Cup
T55	Method for Water in Petroleum Products and Bituminous Materials
T179	Test Method for Effect of Heat and Air on Asphalt Materials (Thin-Film Oven Test)
T201	Kinematic Viscosity of Asphalts
T202	Viscosity of Asphalts by Vacuum Capillary Viscometer
T240	Test Method for Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin Film Oven Test)
TP1	Test Method for Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR)
TP3	Test Method for Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT)
TP5	Test Method for Determining Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

2.2 ASTM Documents:

D8	Standard Definitions of Terms Relating to Materials for Roads and Pavements
D4402	Method for Viscosity Determinations of Unfilled Asphalt Using the Brookfield Thermosel Apparatus

2.3 SHRP Documents:

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[1] *This standard is based on SHRP Product 1001.*

3. Terminology

3.1 Definitions

3.1.1 Definitions for many terms common to asphalt cement are found in ASTM D8.

3.1.2 asphalt binder, n — an asphalt-based cement that is produced from petroleum residue either with or without the addition of non-particulate organic modifiers.

4. Ordering Information - When ordering under this specification, include in the purchase order the performance grade of asphalt binder required from Table 1 (e.g. PG 52-16 or PG 64-34).

4.1 Asphalt binder grades may be selected by following the procedures described in provisional practice PPX, Selection of Asphalt Binders.

5. Materials and Manufacture

5.1 Asphalt cement shall be prepared by the refining of crude petroleum by suitable methods, with or without the addition of modifiers.

5.2 Modifiers may be any organic material of suitable manufacture, used in virgin or recycled condition, and that is dissolved, dispersed or reacted in asphalt cement to enhance its performance.

5.3 The base asphalt binder shall be homogeneous, free from water and deleterious materials, and shall not foam when heated to 175°C.

5.4 The base asphalt binder shall be at least 99.0% soluble in trichloroethylene as determined by AASHTO T44.

5.5 The bending beam rheometer test, TP1, the direct tension test, TP3, and the dynamic shear rheometer test, TP5, are not suitable for asphalt binders in which fibers or other discrete particles are larger than 250 μm in size.

5.6 The grades of asphalt binder shall conform to the requirements given in Table 1.

6. Sampling - The material shall be sampled in accordance with Method T 40.

7. Test Methods - The properties outlined in 5.3, 5.4 and 5.6 shall be determined in accordance with T44, T48, T55, T179, T240, PP1, TP1, TP3, TP5, and ASTM D4402.

8. Inspection and Certification - Inspection and certification of the material shall be agreed upon between the purchaser and the seller. Specific requirements shall be made part of the purchase contract.

9. Rejection and Rehearing - If the results of any test do not conform to the requirements of this specification, retesting to determine conformity is performed as indicated in the purchase order or as otherwise agreed upon between the purchaser and the seller.

10. Key Words - Asphalt binder, asphalt cement, modifier, performance specifications, rheology, direct tension, pressure aging, flash point.

Table 1. Performance Graded Asphalt Binder Specification

PERFORMANCE GRADE	PG 46-			PG 52-							PG 58-					PG 64-					
	34	40	46	10	16	22	28	34	40	46	16	22	28	34	40	10	16	22	28	34	40
Average 7-day Maximum Pavement Design Temperature, °C[a]	<46			<52							<58					<64					
Minimum Pavement Design Temperature, °C[a]	>-34	>-40	>-46	>-10	>-16	>-22	>-28	>-34	>-40	>-46	>-16	>-22	>-28	>-34	>-40	>-10	>-16	>-22	>-28	>-34	>-40
ORIGINAL BINDER																					
Flash Point Temp, T48: Minimum °C	230																				
Viscosity, ASTM D4402:[b] Maximum, 3 Pa•s, Test Temp, °C	135																				
Dynamic Shear, TP5:[c] G*/sinδ, Minimum, 1.00 kPa Test Temp @ 10 rad/s, °C	46			52							58					64					
ROLLING THIN FILM OVEN (T240) OR THIN FILM OVEN RESIDUE (T179)																					
Mass Loss, Maximum, percent	1.00																				
Dynamic Shear, TP5: G*/sinδ, Minimum, 2.20 kPa Test Temp @ 10 rad/s, °C	46			52							58					64					
PRESSURE AGING VESSEL RESIDUE (PP1)																					
PAV Aging Temperature, °C[d]	90			90							100					100					
Dynamic Shear, TP5: G*sinδ, Maximum, 5000 kPa Test Temp @ 10 rad/s, °C	10	7	4	25	22	19	16	13	10	7	25	22	19	16	13	31	28	25	22	19	16
Physical Hardening[e]	Report																				
Creep Stiffness, TP1:[f] S, Maximum, 300 MPa, *m - value, Minimum, 0.300* Test Temp @ 60s, °C	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30
Direct Tension, TP3:[f] Failure Strain, Minimum, 1.0% Test Temp @ 1.0 mm/min, °C	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30

[a] Pavement temperatures are estimated from air temperatures using an algorithm contained in the *SUPERPAVE* software program, may be provided by the specifying agency, or by following the procedures as outlined in PPX.

[b] This requirement may be waived at the discretion of the specifying agency if the supplier warrants that the asphalt binder can be adequately pumped and mixed at temperatures that meet all applicable safety standards.

[c] For quality control of unmodified asphalt cement production, measurement of the viscosity of the original asphalt cement may be substituted for dynamic shear measurements of G*/sinδ at test temperatures where the asphalt is a Newtonian fluid. Any suitable standard means of viscosity measurement may be used, including capillary or rotational viscometry (AASHTO T201 or T202).

[d] The PAV aging temperature is based on simulated climatic conditions and is one of three temperatures 90°C, 100°C or 110°C. The PAV aging temperature is 100°C for PG 64- and above, except in desert climates, where it is 110°C.

[e] Physical Hardening — TP1 is performed on a set of asphalt beams according to Section 13.1, except the conditioning time is extended to 24 hrs ± 10 minutes at 10°C above the minimum performance temperature. The 24-hour stiffness and *m*-value are reported for information purposes only.

[f] If the creep stiffness is below 300 MPa, the direct tension test is not required. If the creep stiffness is between 300 and 600 MPa the direct tension failure strain requirement can be used in lieu of the creep stiffness requirement. The *m*-value requirement must be satisfied in both cases.

Table 1. Performance Graded Asphalt Binder Specification
(Continued)

PERFORMANCE GRADE	PG 70-						PG 76-					PG 82-				
	10	16	22	28	34	40	10	16	22	28	34	10	16	22	28	34
Average 7-day Maximum Pavement Design Temp, °C[b]	<70						<76					<82				
Minimum Pavement Design Temperature, °C[b]	>-10	>-16	>-22	>-28	>-34	>-40	>-10	>-16	>-22	>-28	>-34	>-10	>-16	>-22	>-28	>-34
ORIGINAL BINDER																
Flash Point Temp, T48: Minimum °C	230															
Viscosity, ASTM D4402:[b] Maximum, 3 Pa•s, Test Temp, °C	135															
Dynamic Shear, TP5:[c] $G^*/\sin\delta$, Minimum, 1.00 kPa Test Temp @ 10 rad/s, °C	70						76					82				
ROLLING THIN FILM OVEN (T240) OR THIN FILM OVEN (T179) RESIDUE																
Mass Loss, Maximum, percent	1.00															
Dynamic Shear, TP5: $G^*/\sin\delta$, Minimum, 2.20 kPa Test Temp @ 10 rad/s, °C	70						76					82				
PRESSURE AGING VESSEL RESIDUE (PP1)																
PAV Aging Temperature, °C[e]	100(110)						100(110)					100(110)				
Dynamic Shear, TP5: $G^*\sin\delta$, Maximum, 5000 kPa Test Temp @ 10 rad/s, °C	34	31	28	25	22	19	37	34	31	28	25	40	37	34	31	28
Physical Hardening[a]	Report															
Creep Stiffness, TP1:[f] S, Maximum, 300.0 MPa, *m* - value, Minimum, 0.300 Test Temp @ 60s, °C	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	0	-6	-12	-18	-24
Direct Tension, TP3:[f] Failure Strain, Minimum, 1.0% Test Temp @ 1.0 mm/min, °C	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	0	-6	-12	-18	-24

APPENDIX B

AASHTO Standards for

Effect of Heat and Air on a Moving Film of Asphalt (T 240)

and

Flash and Fire Points by Cleveland Open Cup (T 48)

Standard Method of Test
for

Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin Film Oven Test)

AASHTO DESIGNATION: T 240-87[1]
(ASTM DESIGNATION: D 2872-77)

1. SCOPE

1.1 This test is used to measure the effect of heat and air on a moving film of semi-solid asphaltic materials. The effects of this treatment are determined from measurements of the properties of the asphalt before and after the test.

2. SUMMARY

2.1 A moving film of asphaltic material is heated in an oven for 75 minutes at 163°C (325°F). The amount of hardening is determined from physical tests. An optional procedure is also provided for determining the change in mass.

3. APPARATUS

3.1 *Oven*—This shall be a double-walled electrically heated convection type. Its inside dimensions are height 15 in. (381 mm), width 19 in. (483 mm), and depth (with door closed) 17½ ± ½ in. (445 ± 13 mm). The door shall contain a symmetrically located window with dimensions of 12 in. (305 mm) to 13 in. (330 mm) wide by 8 in. (203 mm) to 9 in. (229 mm) high. The window shall contain two sheets of heat resistant glass separated by an air space. The window should permit an unobstructed view of the interior of the oven. The heating element shall be located below the oven floor and shall be adequate to maintain the required temperature. The oven shall be vented top and bottom. The bottom vents shall be located symmetrically to supply incoming air around the heating elements. They shall have an open area of 2.30 ± 0.24 sq in. (14.8 ± 1.5 cm^2). The top vents shall be symmetrically arranged in the upper part of the oven and have an open area of 1.38 ± 0.09 sq in. (8.9 ± 0.6 cm^2).

3.1.1 The oven shall have an air plenum covering the side walls and ceiling; the air space being 1½ in. (38 mm) deep from the walls and ceiling. At a midpoint in the width of the oven and 6 in. (152 mm) from the face of the circular metal carriage to its axis, a squirrel cage type fan 5¼ in. (133.4 mm) O.D. by 2⅞ in. (73 mm) wide shall be turned at 1,725 RPM by an externally mounted motor. The squirrel cage fan shall be set so that the fan turns in an opposite direction to its vanes. The air flow characteristics of the fan-plenum system shall be suction from the floor of the oven through the wall plenums and exiting of the air through the fan. Figures 1 and 2 show details of this plenum-system.

3.1.2 The oven shall be equipped with a proportional control thermostat capable of maintaining 163°C (325°F) temperature within ± 0.5°C (± 1.0°F). The thermometer shall be hung from or affixed to a mounting in the ceiling which is 2 in. (51 mm) from the right side of the oven at a midpoint in the depth of the oven so that the bulb of the thermometer is within 1 in. (25 mm) of an imaginary line level to the shaft of the circular metal carriage. The heating controls shall be capable of bringing the fully loaded oven back to the test temperature within a 10-minute period after insertion of the samples in a preheated oven.

3.1.3 The oven shall be provided with a 12 in. (305 mm) diameter vertical circular carriage—see Figure 2 for details. This carriage shall be provided with suitable openings and clips for firmly holding eight glass containers—see Figure 3—in a horizontal position. The vertical carriage shall be mechanically driven through a 3/4 in. (19 mm) diameter shaft at a speed of 15 ± 0.2 RPM.

3.1.4 The oven shall be equipped with an air jet positioned to blow heated air into each bottle at its lowest point of travel. The air jet shall have an outlet orifice 0.04 in. (1.02 mm) in diameter (No. 60 drill) connected to a 25 ft (7.6 m) length of 5/16 in. (7.9 mm) O.D. copper tubing. This tubing shall be coiled to lie flat on the bottom of the oven and lead to a source of fresh dried, dust-free regulated air.

NOTE 1—Activated Silica Gel treated with an indicator is a satisfactory desiccant for the dried air.

3.2 *Flow Meter*—The flowmeter may be any suitable type capable of accurately measuring the air flow at a rate of 4,000 ml per minute at the outlet of the copper tube.

3.3 *Thermometer*—This shall be an ASTM Loss on Heat Thermometer 13 C as prescribed in the Standard Specifications for ASTM Thermometers (ASTM E-1).

3.4 *Container*—The container in which the sample is to be tested shall be of heat resistant glass conforming to the dimensions shown in Figure 3.

3.5 *Balance*—If the loss-on-heating is desired, a Class B balance conforming to the requirements of AASHTO M 231 is required. If only the residue is desired, a Class G 2 balance conforming to M 231 may be used.

4. PREPARATION OF OVEN

4.1 Position the air outlet orifice so that it is 1/4 in. (6.4 mm) from the opening of the glass container. The orifice shall

[1] Similar but not technically identical to ASTM method.

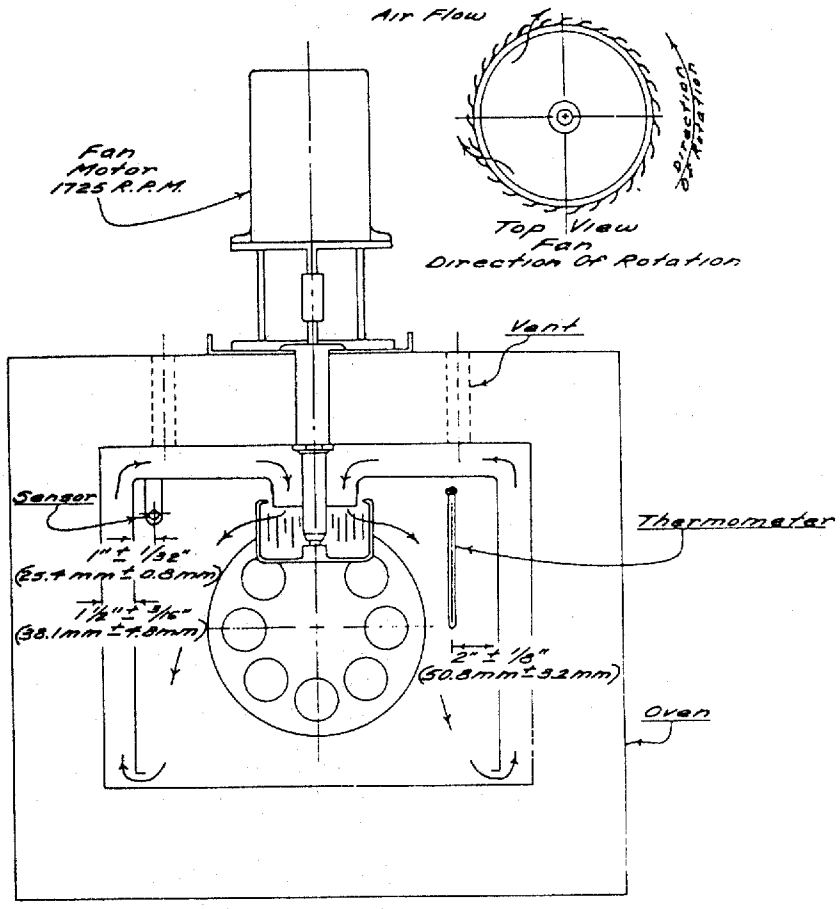


FIGURE 1 Schematic of Air Flow Front View

also be so positioned that the jet blows horizontally into the central arc of the opening of the circling glass container.

4.2 Position the thermometer specified in 3.3 so that the end of the bulb of the thermometer is within 1 in. (25 mm) level to the center of the shaft holding the revolving carriage.

4.3 Level the oven so that the horizontal axes of the glass containers are level when in position in the carriage.

4.4 Preheat the oven for a minimum of 16 h prior to testing with the controls on the setting which will be used during the operation of the oven. The control thermostat shall be adjusted so that when the oven is fully loaded and the air is on, it will return to 163 ± 0.5 C (325 ± 1.0 F) within the 10-min warm-up period.

5. PROCEDURE

5.1 The sample as received shall be free of water. Heat the sample in its container with a loosely fitted cover in an oven not to exceed 163 C (325 F) for the minimum time necessary to insure that the sample is completely fluid. Manually stir the sample but avoid incorporating air bubbles.

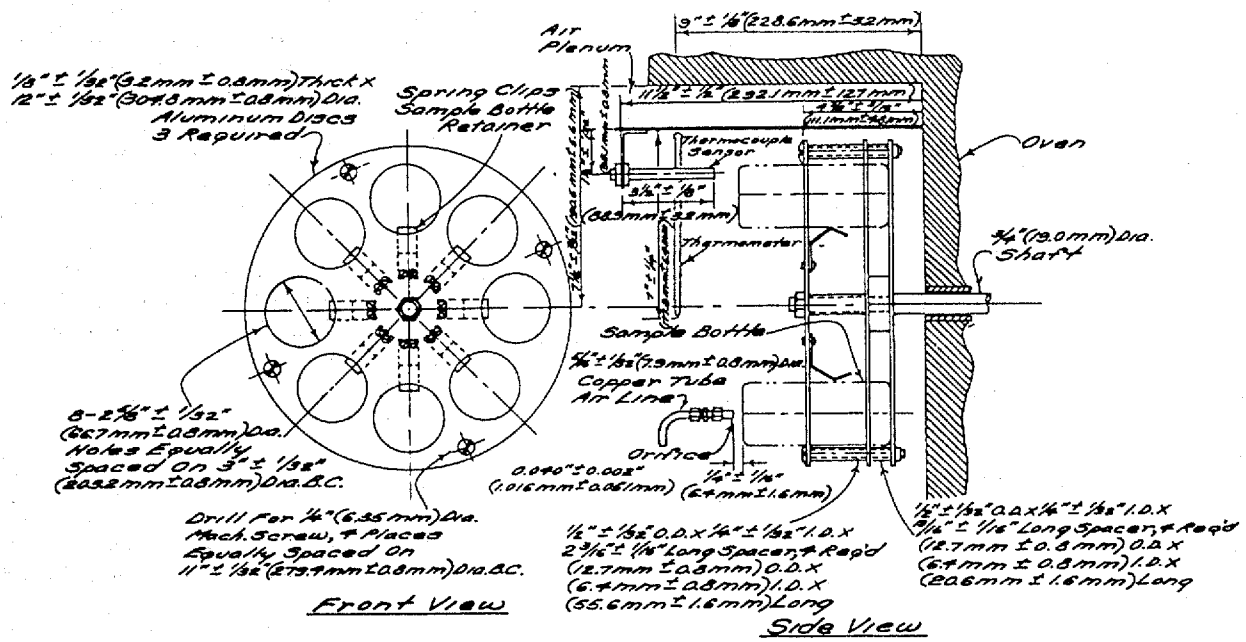


FIGURE 2 Circular Metal Carriage

5.2 Pour 35 ± 0.5 grams of the sample into each of the required glass containers providing sufficient material for characterizing tests which are to be run on the residue.

NOTE 2—For referee testing, eight (8) glass containers of the sample will be required.

5.3 When the change in mass is not required, allow the container to cool to approximately room temperature before placing in the oven as directed in 5.4. When the quantitative value of the change is desired, two separate bottles should be used for the determination. Cool the bottles for the test to room temperature and weigh each bottle separately to the nearest 0.001 g.

NOTE 3—Do not use the residue from change in mass determination for other tests.

5.4 With the oven at operating temperature, arrange the containers holding the asphalt in the carriage so that the carriage is balanced. Fill any unused spaces in the carriage with empty containers. Close the door and rotate the carriage assembly at a rate of 15 ± 0.2 RPM. Start the air flow at a set rate of 4000 ± 200 ml per minute corrected to standard barometric pressure and temperature. Maintain the samples in the oven with the air flowing and the carriage rotating for 85 minutes. The test temperature 163 ± 0.5°C (325 ± 1°F) shall be reached within the first 10 minutes—otherwise discontinue the test. At the conclusion of the testing period remove the containers from the oven. If the change in mass is not being determined, proceed in accordance with 5.5. For the glass containers on which the change in mass is being determined, cool to room temperature in a desiccator, then weigh to the nearest 0.001 g and calculate the change in mass on the basis of the asphalt in the container. Discard the residue.

5.5 Immediately pour all of the residue, without scraping, from each bottle into a container large enough so that when all of it is collected the container is not over 75 percent full. Do not let the moving film bottles cool nor should the bottles be reheated to obtain more residue. Proceed as described in 5.6.

5.6 Test the residue within 24 hours of performing the moving film test.

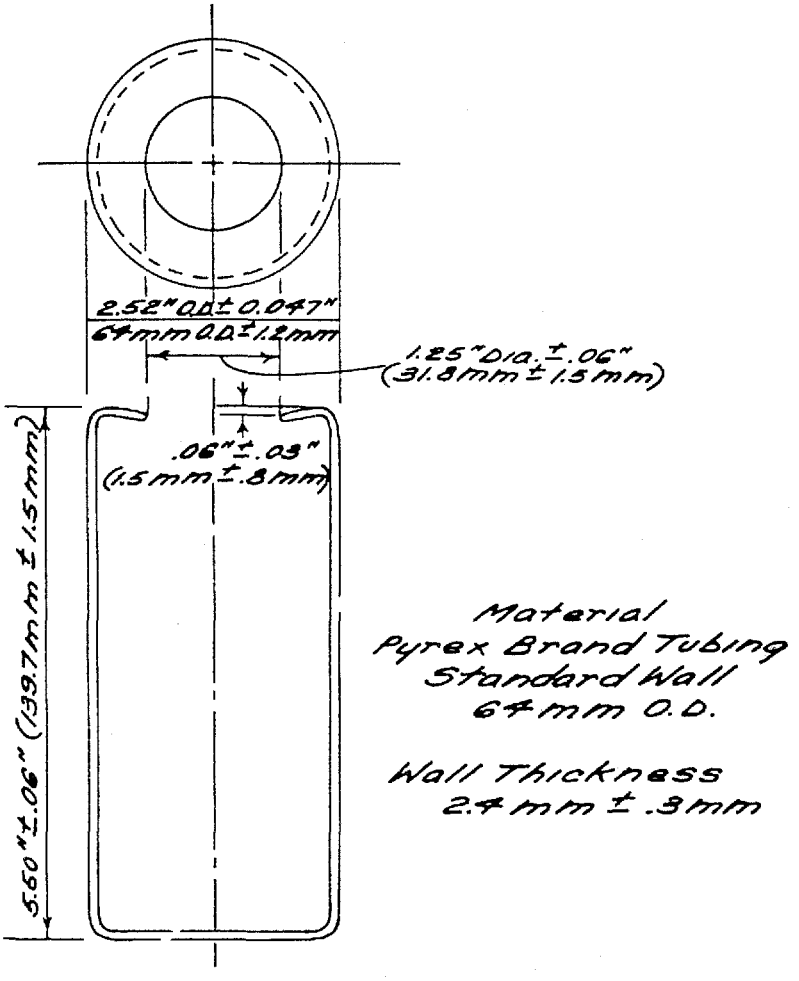


FIGURE 3 Sample Bottle

6. REPORT

6.1 Report the results from the moving film test in terms of the physical changes in the asphalt brought about by this method. These values are obtained by performing appropriate tests on the asphalt before and after the moving film oven cycle.

7. PRECISION

7.1 The single-operator coefficient of variation for viscosity tests at 60 C (140 F) on the residue after heating has been found to be 2.3 percent (1S%). Therefore such viscosity results for two properly conducted tests by the same operator should not differ by more than 6.5 percent of their average (D2S%).

7.2 The multilaboratory coefficient of variation for viscosity tests at 60 C (140 F) on the residue after heating has been found to be 4.2 percent (1S%). Therefore results of such viscosity tests by two different laboratories on samples of the same material should not differ by more than 11.9 percent of their average (D2S%).

7.3 The precision of loss of weight determinations or of tests on the residues after heating other than viscosity have not been determined.

Standard Method of Test
for

Flash and Fire Points by Cleveland Open Cup

AASHTO DESIGNATION: T 48-89[1]
(ASTM DESIGNATION: D 92-85)

1. SCOPE

1.1 This method covers the determination of the flash and fire points, by Cleveland Open Cup Tester, of petroleum products and other liquids, except fuel oils and those materials having an open cup flash point below (79 C) 175 F as determined by the Cleveland Open Cup Tester.

NOTE 1—It is the practice in the United Kingdom and in many other countries to use IP Method 35, Flash Point (Open) and Fire Points by Means of the Pensky-Martens Apparatus unless AASHTO T 73. Test for Flash Point by Pensky-Martens Closed Tester is specified. This method may occasionally be specified for the determination of the fire point of a fuel oil. For the determination of the flash points of fuel oils, use AASHTO T 73 IP 34, AASHTO T 73 should also be used when it is desired to determine the possible presence of small but significant concentrations of lower flash point substances which may escape detection by T 48. AASHTO T 79, Flash Point With Tag Open-Cup Apparatus, may be employed if the flash point is below 79 C (175 F) as determined by T 48.

1.2 "This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use."

2. REFERENCED DOCUMENTS

2.1 *AASHTO Standards:*
T 73 Flash Point by Pensky-Martens Closed Tester
T 79 Flash Point with Tag Open-Cup Apparatus

2.2 *ASTM Standard:*
E.1 Specification for ASTM Thermometers

2.3 *Other Method:*
IP Method 35 Flash Point (Open) and Fire Point by Means of the Pensky-Martens Apparatus

3. DEFINITIONS

3.1 *Flash*—point the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of a test flame causes the vapor of a specimen to ignite under specified conditions of test.

NOTE 2—The material is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the specimen.

Occasionally, particularly near the actual flash point, the application of the test flame will cause a blue halo or an enlarged flame; this is not a flash and should be ignored.

3.2 *Fire Point*—the lowest temperature at which a specimen will sustain burning for 5 s.

4. SUMMARY OF METHOD

4.1 The test cup is filled to a specified level with the sample. The temperature of the sample is increased rapidly at first and then at a slow constant rate as the flash point is approached. At specified intervals a small test flame is passed across the cup. The lowest temperature at which application of the test flame causes the vapors above the surface of the liquid to ignite is taken as the flash point. To determine the fire point, the test is continued until the application of the test flame causes the oil to ignite and burn for at least 5 s.

5. SIGNIFICANCE AND USE

5.1 Flash point measures the tendency of the sample to form a flammable mixture with air under controlled laboratory conditions. It is only one of a number of properties that must be considered in assessing the overall flammability hazard of a material.

5.2 Flash point is used in shipping and safety regulations to define "flammable" and "combustible" materials. One should consult the particular regulation involved for precise definitions of these classes.

5.3 Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable material.

5.4 Fire point measures the characteristics of the sample to support combustion.

6. APPARATUS

6.1 *Cleveland Open Cup Apparatus*—This apparatus consists of the test cup, heating plate, test flame applicator, heater, and supports as described in detail in the appendix. One form of the assembled apparatus, the heating plate, and the cup are illustrated in Figures 1, 2, and 3, respectively.

6.2 *Shield*—A shield 460 mm (18 in.) square and 610 mm (24 in.) high and having an open front is recommended.

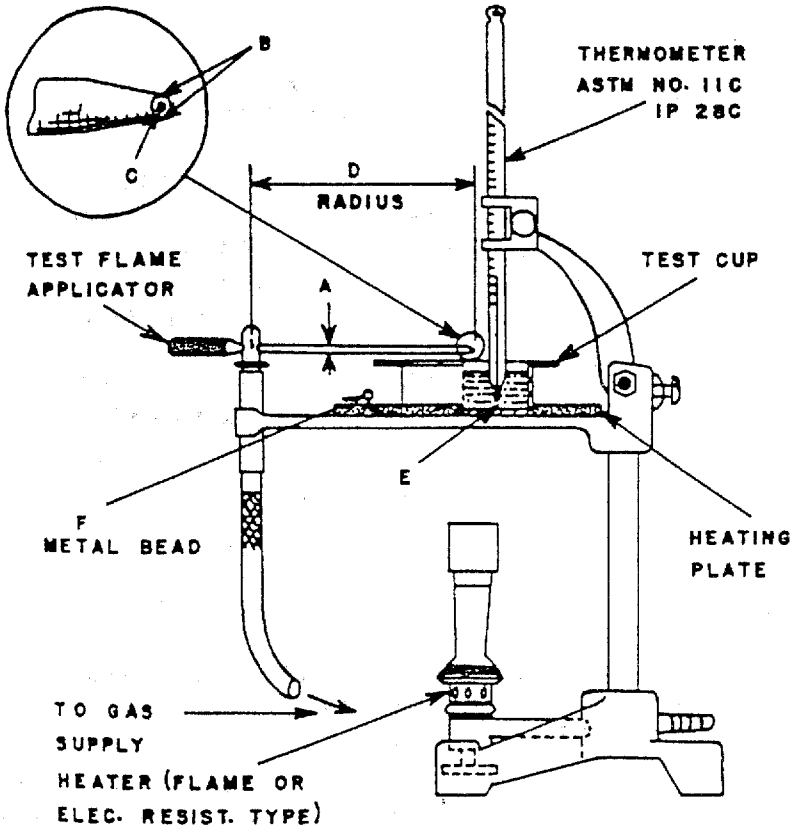
6.3 *Thermometer*—A thermometer having a range as shown below and conforming to the requirements as prescribed

[1] Similar but not technically identical to ASTM method.

in ASTM Specification E1 or in the Specifications for IP Standard Thermometers.

Temperature Range	Thermometer Number ASTM	IP
20 to 760 F	11 F	28 F
− 6 to + 400 C	11C	28 C

NOTE 3—There are automatic flash point testers available and in use which may be advantageous in the saving of testing time, permit the use of smaller samples, and have other factors which may merit their use. If automatic testers are used, the user must be sure that all of the manufacturer's instructions for calibrating, adjusting, and operating the instrument are followed, in any cases of dispute, the flash point as determined manually shall be considered the referee test.



	MILLIMETRES MIN	MAX	INCHES MIN	MAX
A - DIA. OF APPLICATOR	-	5.0	-	0.20
B - DIA. OF TIP	1.6	5.0	0.06	0.20
C - DIA. OF ORIFICE	(0.8 APPROX.)		(0.031 APPROX.)	
D - RADIUS OF SWING	150	-	6	-
E - INSIDE BOTTOM OF CUP TO BOTTOM OF THERMOMETER	(6.4 APPROX.)		(0.25 APPROX.)	
F - DIA. OF OPTIONAL COMPARISON BEAD	3.8	5.4	0.15	0.21

FIGURE 1 Cleveland Open Cup Apparatus

7. SAFETY PRECAUTIONS

7.1 The operator must exercise and take appropriate safety precautions during the initial application of the test flame, since samples containing low-flash material may give an abnormally strong flash when the test flame is first applied.

8. SAMPLING

8.1 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily and make a transfer unless the sample temperature is at least the equivalent of 18°F (10°C) below the expected flash point. Do not use samples from leaky containers for this test.

8.2 Do not store samples in plastic (polyethylene, polypropylene, etc.) containers, since volatile material may diffuse through the walls of the enclosure.

8.3 Light hydrocarbons may be present in the form of gases, such as propane or butane and may not be detected by testing because of losses during sampling and loading of the test apparatus. This is especially evident on heavy residuums or asphalts from solvent extraction processes.

9. PREPARATION OF APPARATUS

9.1 Support the apparatus on a level steady table in a draft-free room or compartment. Shield the top of the apparatus from strong light by any suitable means to permit ready detection of the flash point. Tests in a laboratory hood (Note 4) or any location where drafts occur are not to be relied upon.

NOTE 4—With some samples whose vapors or products of pyrolyica are objectionable, it is permeable to place the apparatus with a shield in a hood, the draft of which is adjustable so that vapors may be withdrawn without causing air current over the last cup during the final 36 C (100 F) rise in temperature prior to the flash point.

9.2 Wash the test cup with an appropriate solvent to remove any oil or traces of gum or residue remaining from a previous test. If any deposits of carbon are present, they should be removed with steel

wool. Flush the cup with cold water and dry for a few minutes over an open flame, on a hot plate, or in an oven to remove the last traces of solvent and water. Cool the cup to at least 56 C (100 F) below the expected flash point before using.

9.3 Support the thermometer in a vertical position with the bottom of the bulb 6.4 mm (1/4 in.) from the bottom of the cup and located at a point halfway between the center and side of the cup on the diameter perpendicular to the arc (or line) of the sweep of the test flame and on the side opposite to the test flame burner arm.

10. PROCEDURE

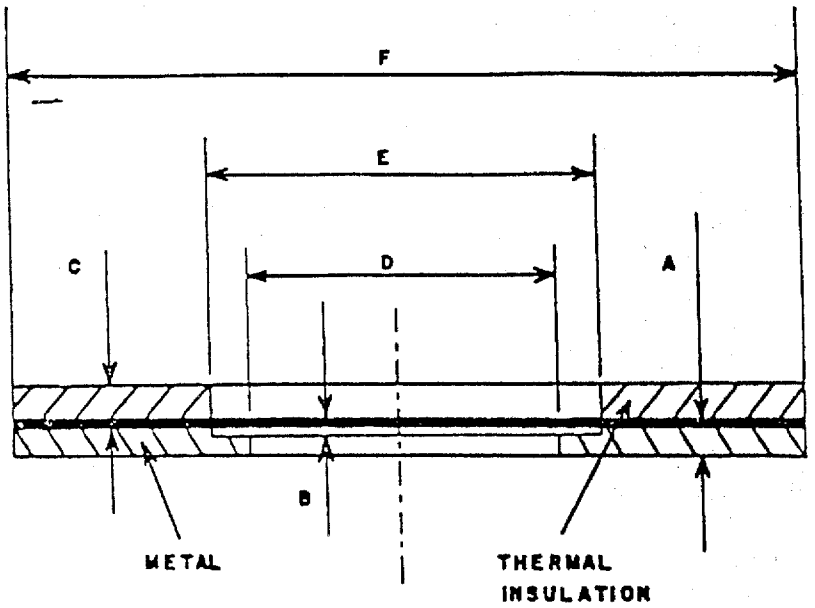
10.1 Fill the cup, at any convenient temperature (Note 5) not exceeding 100 C or 180 F above the softening point, so that the top of the meniscus is at the filling line. To aid in this operation, a Filling, Level Gage (A7) may be used. If too much sample has been added to the cup, remove the excess, using a pipet or other suitable device; however, if there is sample on the outside of the apparatus, empty, clean, and refill it. Destroy any air bubbles on the surface of the sample (Note 6).

NOTE 5—Viscous samples should be heated until they are reasonably fluid before being poured into the cup. For asphalt cement, the temperature during heating must not exceed 100 C or 180 F above the expected softening point. Extra caution must be exercised with liquid asphalts which should be heated only to the lowest temperature at which they can be poured.

NOTE 6—The sample cup may be filled away from the apparatus provided the thermometer is preset with the cup in place and the sample level is correct at the beginning of the test. A shim 6.4 mm (1/4 in.) thick is useful in obtaining the correction distance from the bottom of the bulb to the bottom of the cup.

10.2 Light the test flame and adjust it to a diameter of 3.8 to 5.4 mm (0.15 to 0.21 in.).

10.3 Apply heat initially so that the rate of temperature rise of the sample is 14 to 17 C (25 to 30 F) per minute. When the sample temperature is approximately 56 C (100 F) below the anticipated flash point, decrease the heat so that the rate of temperature rise for the 28 C (50 F) before the flash point is 5 to 6 C (9 to 11 F) per minute.



	MILLIMETRES		INCHES	
	MIN	MAX	MIN	MAX
A	6.4 NOMINAL		0.25 NOMINAL	
B	0.5	1.0	0.02	0.04
C	6.4 NOMINAL		0.25 NOMINAL	
D- DIAMETER	54.5	56.5	2.15	2.22
E- DIAMETER	69.5	70.5	2.74	2.78
F- DIAMETER	150 NOMINAL		6 NOMINAL	

FIGURE 2 Heating Plate

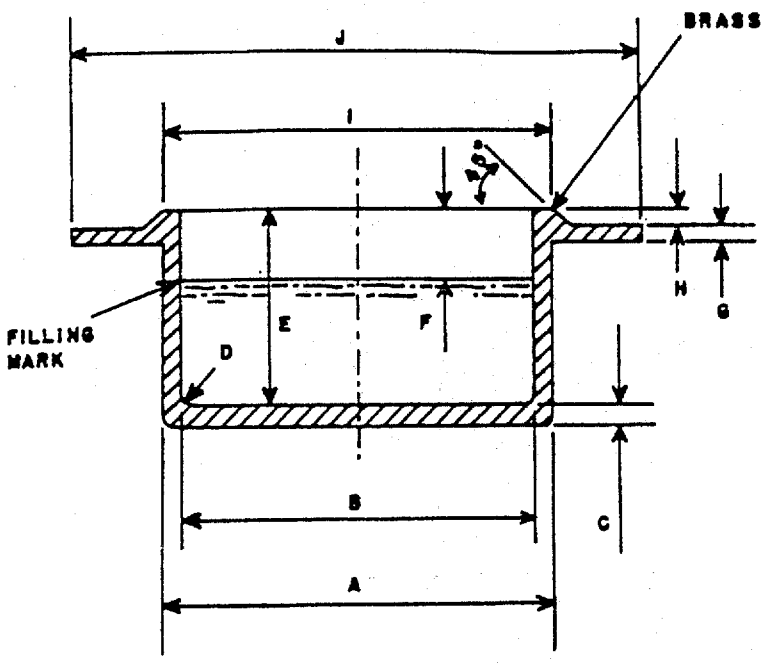
10.4 Starting at least 28 C (50 F) below the flash point, apply the test flame when the temperature read on the thermometer reaches each successive 2 C (5 F) mark. Pass the test flame across the center of the cup, at right angles to the diameter which passes through the thermometer. With a smooth, continuous motion apply the flame either in a straight line or along the circumference of a circle having a radius of at least 150 mm or 6 in. The center of the test flame must move in a plane not more than 2.5 mm or 0.10 in. above the plane of the upper edge of the cup passing in one direction first, then in the opposite direction the next time. The time consumed in passing the test flame across the cup shall be about 1 s. During the last 17 C (30 F) rise in temperature prior to the flash point, care must be taken to avoid disturbing the vapors in the test cup by careless movements or breathing near the cup.

NOTE 7—If a skin should form between the flash or fire point is reached, move it carefully aside with a small spatula or stirring rod and continue the determination.

NOTE 8—Caution—Meticulous attention to all details relating to the test flame application, size of the test flame, rate of temperature increase, and rate of passing the test flame over the sample is necessary for good results.

10.5 Record as the observed flash point the temperature read on the thermometer when a flash appears at any point on the surface of the oil, but do not confuse the true flash with the bluish halo that sometimes surrounds the test flame.

10.6 To determine the fire point, continue heating so that the sample temper-



	Millimetres		Inches	
	Min	Max	Min	Max
A	67.5	69	2.66	2.72
B	62.5	64.0	2.46	2.52
C	2.8	3.6	0.11	0.14
D-Radius	4 Approx.		0.16 Approx.	
E	32.5	34	1.28	1.34
F	9	10	0.35	0.39
G	1.8	3.4	0.07	0.13
H	2.8	3.6	0.11	0.14
I	67	70	2.60	2.75
J	97	101	3.8	4.0

FIGURE 3 Cleveland Open Cup

ture increases at a rate of 5 to 6 C (9 to 11 F). Continue the application of the test flame at 2 C (5 F) intervals until the oil ignites and continues to burn for at least 5 s. Record the temperature at this point as the fire point of the oil.

11. CALCULATION AND REPORT

11.1 Observe and record the barometric pressure at the time of the test. When the pressure differs from 760 mm Hg, correct the flash or fire point, or both, by means of the following equations:

Corrected flash or fire point, or both = F + 0.06 (760 − P) or Corrected flash or fire point, or both = C + 0.03 (760 − P) where:

F = observed flash or fire point, or both, to the nearest 5°F,

C = observed flash or fire point, or both, to the nearest 2°C, and

P = barometric pressure, mm Hg.

11.2 Record the corrected flash or fire point, or both, to the nearest 5°F or 2°C.

11.3 Report the recorded flash or fire point value, or both, as the COC flash or fire point, or both, T 48-IP 36 of the sample tested.

12. PRECISION

12.1 The following data should be used for judging the acceptability of results (95 percent confidence)

12.1.1 Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

	Repeatability
Flash point	8C (15F)
Fire point	8C (15F)

12.1.2 The result submitted by each of two laboratories should be considered suspect if the results differ by more than the following amounts:

	Reproducibility
Flash point	17 C (30 F)
Fire point	14 C (25 F)

APPENDIX

APPARATUS

The Cleveland open tester shall consist of a test cup, heating plate, test flame applicator, heater, thermometer support, and heating plate support, all confronting to the following requirements:

A1. *Test Cup*—of brass conforming to the dimensional requirements shown in Figure 3. The cup may be equipped with a handle.

A2. *Heating Plate*—A brass, cast iron, wrought iron, or steel plate with a center hole surrounded by an area of plane depression, and a sheet of hard asbestos board which covers the metal plate except over the area of plane depression in which the test cup is supported. The essential dimensions of the heating plate are shown in Figure 2; however, it may be square instead of round, and the metal plate may have suitable extensions for mounting the test flame applicator device and the thermometer support. The metal bead, as mentioned in Paragraph A3, may be mounted on the plate so that it extends through and slightly above a suitable small hole in the asbestos board.

NOTE 9—The sheet of hard asbestos board or other heat resistant material which covers the heating plate may be extended beyond the edge of the heating plate to reduce drafts around the

cup. The F dimension given is intended for gas apparatus. For electrically heated apparatus the plate shall be of sufficient size to cover the top of the heater.

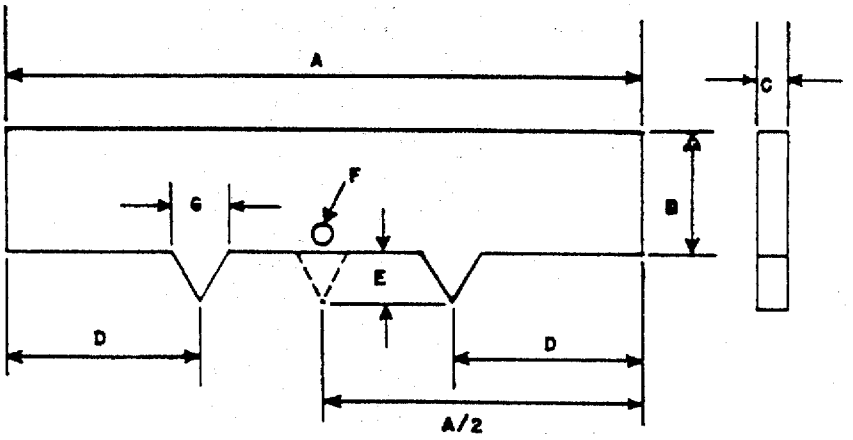
A3. *Test Flame Applicator*—The device for applying the test flame may be of any suitable design, but the tip shall be 1.6 to 5.0 mm or 0.06 to 0.20 in. in diameter at the end and the orifice shall have an approximate diameter of 0.8 mm or 0.031 in. The device for applying the test flame shall be so mounted to permit automatic duplication of the sweep of the test flame, the radius of swing being not less than 150 mm or 6 in. and the center of the orifice moving in a plane not more than 2.5 mm or 0.10 in. above the cup. A bead having a diameter of 3.8 to 5.4 mm or 0.15 to 0.21 in. may be mounted in a convenient position on the apparatus so the size of the test flame can be compared to it.

A4. *Heater*—Heat may be supplied from any convenient source. The use of a gas burner or alcohol lamp is permitted, but under no circumstances are products of combustion or free flame to be allowed to come up around the cup. An electric heater controlled by a variable voltage transformer is preferred. The source of heat shall be centered under the opening of the heating plate with no local superheating. Flame-type heaters may be protected from drafts or excessive radiation by any suitable type of shield that does not project above the level of the upper surface of the asbestos board.

A5. *Thermometer Support*—Any convenient device may be used which will hold the thermometer in the specified position during a test and which will permit easy removal of the thermometer from the test cup upon completion of a test.

A6. *Heating Plate Support*—Any convenient support which will hold the heating plate level and steady may be employed.

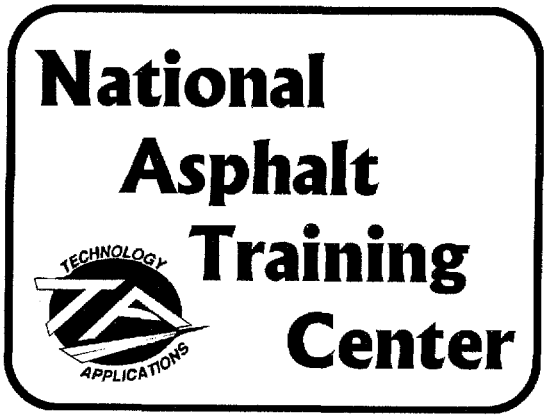
A7. *Filling Level Gage*—A device to aid in the proper adjustment of the sample level in the cup. It may be made of suitable metal with at least one projection, but preferably two for adjusting the sample level in the test cup to 9 to 10 mm (0.35 to 0.39 in.) below the top edge of the cup. A hole 0.8 mm (1/32 in.) in diameter, the center of which is located not more than 2.5 mm or 0.10 in. above the bottom edge of the gage, shall be provided for use in checking the center position of the orifice of the test flame applicator with respect to the rim of the cup. (Figure 4 shows a suitable version.)



	MILLIMETRES	INCHES	
A	100	4	NOMINAL
B	20	3/4	NOMINAL
C	3.2	1/8	NOMINAL
D	30	1-1/4	NOMINAL
E	9-10	0.35-0.39	
F	0.8 DIA. (2.5 MM ABOVE BOTTOM EDGE)	1/32 DIA. (0.10 IN. ABOVE BOTTOM EDGE)	NOMINAL MAXIMUM
G	10	3/8	NOMINAL

FIGURE 4 Filling Level Gage

APPENDIX C



Forms for Recording Binder Test Results

National Asphalt Training Center

PROJECT: _____ COMMENTS/REQUEST: _____

SAMPLE: _____

TECH: _____

DATE: _____ PERFORMANCE GRADE: _____

PERFORMANCE GRADE	PG 52							PG 58					PG64					PG 70					
	-10	-16	-22	-28	-34	-40	-46	-16	-22	-28	-34	-40	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-40
ORIGINAL BINDER																							
FLASH POINT TEMP Min 230 C																							
ROTATIONAL VISC Max, 3 Pa.s (3000cP) Test Temp, 135 C																							
DYNAMIC SHEAR G*/sin delta, Min 1.00 kPa Test Temp @ 10 rad/s, C	52							58					64					7 0					
RTFO RESIDUE Percent change, 1.00 Max loss																							
DYNAMIC SHEAR G*/sin delta, Min 2.20 kPa Test Temp @ 10 rad/s, C	52							58					64					7 0					
PAV AGING 20 hrs @ 2.07 MPa	90							100					100					100/(110)					
DYNAMIC SHEAR G*sin delta, Max 5,000 kPa Test Temp @ 10 rad/s, C	25	22	19	16	13	10	7	25	22	19	16	13	28	25	22	19	16	34	31	28	25	22	19
CREEP STIFFNESS S, Max 300.0 MPa STIFFNESS Test Temp @ 60s, C m-value, Min 0.300 m-VALUE	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30
PHYSICAL HARDENING 24 Hours Conditioning S= m=																							
DIRECT TENSION Failure Strain, Min 1.0% Test Temp @ 1.0 mm/min, C	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30

Rotational Viscosity:

Test Temperature: _____ C Spindle #: _____ Speed: _____ RPM

Three Readings/One Minute Intervals: _____ , _____ , _____

Average: _____ cP X .001 = [Pa.S]

Note: 1cP = 0.001 Pa.s

Rolling Thin Film Oven Residue:

Time in oven: _____ (+85 minutes) Time out of oven: _____

Bottle number:		
Weight of Bottle and asphalt:	_____ g	_____ g
Weight of Bottle:	_____ g	_____ g
Weight of Asphalt before heating:	_____ g	_____ g
Asphalt and bottle after heating:	_____ g	_____ g
Mass loss (-) or gain (+):	_____ g	_____ g
Percent loss (-) or gain (+):	_____ %	_____ %

Average percent loss or gain: [%]

Pressure Aging Vessel Residue:

Time In: _____ (+ 20 Hours) Time Out: _____

Aging Test Temperature, Nearest 0.1 C: _____

Maximum and Minimum aging temperature recorded, nearest 0.1 C:

Max: _____

Min: _____

Total time during aging that temperature was ouside the specified range, nearest 0.1 minute: _____

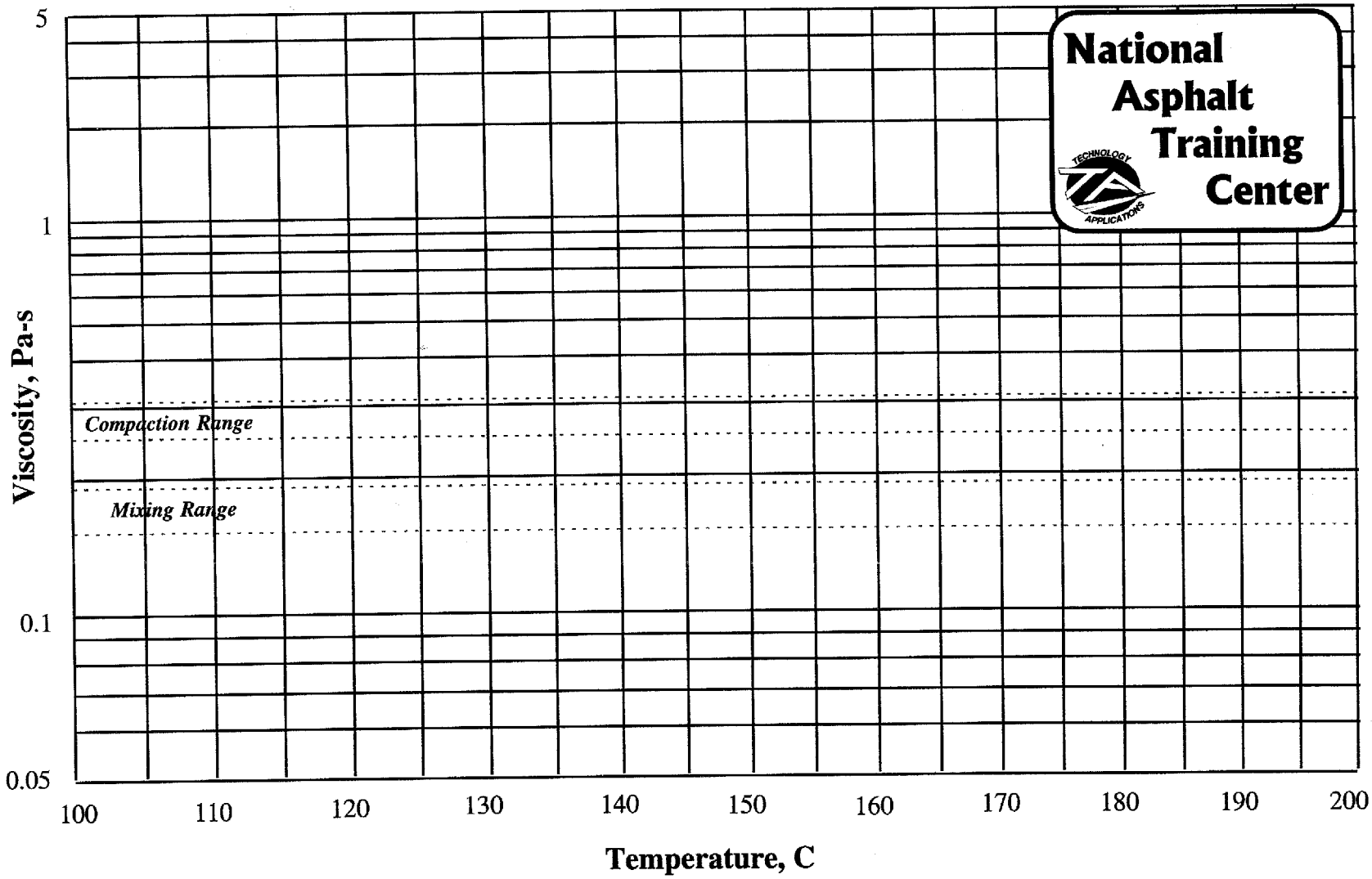
Total aging time, hours and minutes: _____

Bending Beam Rheometer:

Sample ID:					
Time Poured:					
Time Trimmed:					
Time In Bath:					
Time Tested:					

Direct Tension Time:

Sample ID:					
Time Poured:					
Time Trimmed:					
Time In Chamber:					
Time Tested:					


National
Asphalt
Training
Center
TECHNOLOGY
APPLICATIONS
5
1
0.1
0.05
Viscosity, Pa-s
Compaction Range
Mixing Range
100
110
120
130
140
150
160
170
180
190
200
Temperature, C

Printed By
Federal Highway Administration
U.S. Department of Transportation
400 Seventh Street, SW
Washington, D.C. 20590
HTA-21/8-94(2M)QE