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Designation: D 6723 – 01

# Standard Test Method for Determining the Fracture Properties of Asphalt Binder in Direct Tension (DT)<sup>1</sup>

This standard is issued under the fixed designation D 6723; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

# 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 can be used with unaged material or with material aged using Test Method D 2872 (RTFOT), Practice D 6521 (PAV), or AASHTO T240 (RTFOT) and AASHTO PP1 (PAV). The test apparatus is designed for testing within the temperature range from +6 to -36°C.

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

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

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

# 2. Referenced Documents

2.1 ASTM Standards:

- C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>
- D 8 Terminology Relating to Materials for Roads and Pavements<sup>3</sup>
- D 140 Practice for Sampling Bituminous Materials<sup>3</sup>
- D 2872 Test Method for Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test)<sup>3</sup>
- D 6373 Specification for Performance Graded Asphalt Binder<sup>3</sup>
- D 6521 Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)<sup>3</sup>
- E 1 Specification for ASTM Thermometers<sup>4</sup>
- E 4 Practices for Force Verification of Testing Machines<sup>5</sup>
- E 77 Test Method for the Inspection and Verification of Thermometers  $^{4}$

- <sup>2</sup> Annual Book of ASTM Standards, Vol 04.02.
- <sup>3</sup> Annual Book of ASTM Standards, Vol 04.03.
- <sup>4</sup> Annual Book of ASTM Standards, Vol 14.03.

E 83 Practice for Verification and Classification of Extensometers<sup>5</sup>

- E 220 Test Method for Calibration of Thermocouples by Comparison Techniques<sup>4</sup>
- 2.2 Deutche Industrie Norm (DIN) Standard:
- 43760 Standard for Calibrating Thermocouples<sup>6</sup>
- 2.3 AASHTO Standards:
- T 240<sup>7</sup>
- PPI (PAV)<sup>7</sup>

# 3. Terminology

3.1 *Definitions*—For definitions of general terms used in this standard, refer to Terminology D 8.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *brittle*, *adj*—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*—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*—type of failure in a direct tension test where the specimen does not rupture but fails by flow at large strains.

3.2.4 effective gage length, n—for specimens used in this test, the effective gauge length,  $L_e$ , has been determined to be 33.8 mm. This is an effective gauge length that represents the portion of the specimen that contributes to the majority of the strain.

3.2.5 *failure*, *n*—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 strain*, *n*—the tensile strain corresponding to the failure stress.

3.2.7 *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.

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<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.44 on Rheological Tests.

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<sup>&</sup>lt;sup>5</sup> Annual Book of ASTM Standards, Vol 03.01.

<sup>&</sup>lt;sup>6</sup> Deutsches Institut fur Normung e.V. (German Standards Institute), Beuth Verlag GmbH, Burggrafenstrasse 6, 10787, Berlin Germany.

<sup>&</sup>lt;sup>7</sup> Available from the American State Highway and Transportation Officials, 444 N. Capitol Street, N.W., Washington, DC 20001.

3.2.8 gage section, n—the central portion of the specimen where the cross-section does not change with length. For the geometry used in this test method, the gage section is 18 mm in length (see Fig. 1).

3.2.9 *tensile strain*, n—axial strain resulting from the application of a tensile load and calculated as the change in length of the effective gage length caused by the application of the tensile load divided by the original unloaded effective gage length.

3.2.10 *tensile stress*, *n*—axial stress resulting from the application of a tensile load and calculated as the tensile load divided by the original area of cross-section of the specimen.

## 4. Summary of Test Method

4.1 This test method describes the procedure used to measure the stress at failure and strain 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 end tabs are used to bond the asphalt binder during the test and to transfer the tensile load from the test machine to the asphalt binder.

4.2 This test method 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 method is not applicable at temperatures where failure is by ductile flow.

4.3 A displacement transducer is used to measure the elongation of the test specimen as it is pulled in tension at a constant rate of 1.00 mm/min. The load developed during the test is monitored and the tensile strain and stress in the test specimen when the load reaches a maximum are reported as the failure strain and failure stress, respectively.

#### 5. Significance and Use

5.1 Stress at failure is used in a mechanistic pavement cracking model to compute critical cracking temperature. The procedure to compute critical cracking temperature is described in Practice D 6521. The critical cracking temperature is then used in specifying the low temperature grade of asphalt binder in accordance with Specification D 6373.

5.2 The test method is designed to measure the strength of the asphalt binder at the critical cracking temperature. The asphalt binder has limited ability to resist stress without cracking. In the asphalt binder Specification D 6373, failure stress is used to determine the critical cracking temperature.

5.3 For evaluating an asphalt binder for conformance to Specification D 6373, the elongation rate of the gage section is 1.0 mm/min and the test temperature is selected from Table number 1 of Specification D 6373 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 closed feed-back loop displacement-controlled tensile loading machine, (2) a specimen gripping system, (3) either a fluid bath or an insulated chamber for

reliable, accurate and uniform temperature control during testing and conditioning of specimen, (4) real-time load measuring and recording devices, (5) real-time elongation measuring and recording devices, (6) a real-time temperature detection and recording device, and (7) real-time data acquisition and display devices. The system shall have an electromechanical or servo-hydraulic loading unit capable of applying and measuring tension and compression forces of at least 500 N and actuator travel of 20 mm. The system stiffness shall be at least 3 MN/m including the load cell and the loading pins. The unit shall have a transducer to measure and control grip separation and provide a feedback for strain control with a displacement resolution of 1.0 µm. The system shall be capable of closed loop elongation rate control accurate to at least 1 % of the commanded specimen elongation rate using feedback from a displacement transducer mounted between the loading pins or a non-contact extensometer measuring the elongation of the specimen.

6.1.1 Tensile Loading Machine Equipped With Temperature Control-A tensile loading machine with a controlleddisplacement loading frame capable of producing at least a 500.00 N load is required. The loading frame shall be table mounted. The gripping system (loading pins and platens) shall be completely submerged under the cooling fluid if a fluid based system is used. The gripping system shall be a minimum of 25 mm under the cooling fluid surface. Loading shall be accomplished by pulling directly in tension in the plane of the specimen. The distance between the load frame's loading points (between loading pins) shall accommodate specimens with total length (including the end tabs) of at least 100.0 mm, (see Fig. 1). If an air cooled system is used, the testing frame shall be equipped with two standards (columns) with sufficient clear space between the standards so that an insulated temperature control chamber can be placed between the standards.

6.1.2 Specimen Gripping System—The gripping system must produce a self-aligning uniaxial test load and accept the end tabs described in 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 loading platens of the testing machine. Fig. 2 shows a typical grip and loading pin assembly. One grip shall be fixed and remain stationary during the test while the other grip is displaced at the desired elongation rate.

6.1.2.1 Specimen End Tabs—End tabs made from Phenolic G-10 material having the dimensions specified in Figs. 3-5 shall be bonded to both ends of the test specimen to transfer the tensile load to the asphalt binder. The end tabs shall be machined from standard G-10 Phenolic sheet. Each end tab shall contain a precisely machined hole lined with a 304 stainless steel ring. The diameter of the lined hole shall be 10.00  $\pm$  0.05 mm. Gripping of the specimen is accomplished through the bond (adhesion) between the asphalt binder test specimen and the end tab. Each end tab shall be mounted on a specially shaped pin that is part of the gripping system. The specimen shall be mounted on the grips by positioning the end tabs in the test machine such that the end tabs fit onto the pins and are indexed against the face of the grips. Matching the

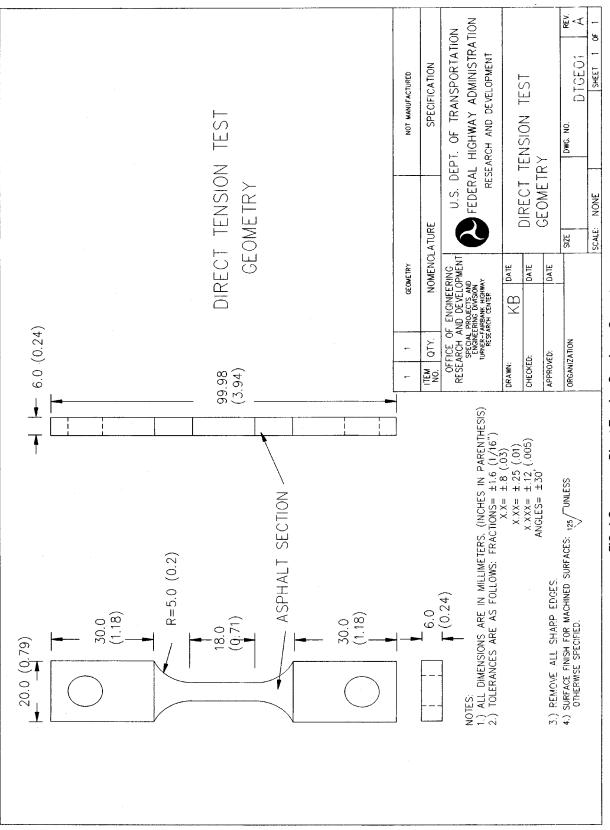
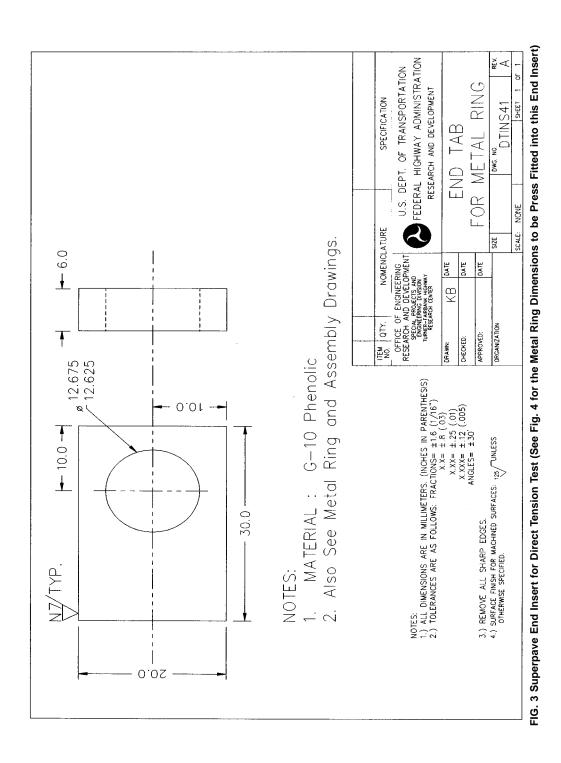


FIG. 1 Superpave Direct Tension Specimen Geometry

FEDERAL HIGHWAY ADMINISTRATION Þ¢ P. U.S. DEPT. OF TRANSPORTATION RESEARCH AND DEVELOPMENT SHEET 1 SPECIFICATION DTLPG1 316 STAINLESS STEEL જે DWG. NO. <u>Z</u> LOADING ├---------SCALE: NONE 2 NOMENCLATURE PRESS SIZE OFFICE OF ENGINEERING RESEARCH AND DEVELOPMENT SPEIAL PROJECTS AND ENGINEERING DIVISION TUNKEERIC DIVISION TUNKEERIC DIVISION DATE DATE DATE GRIP  $\stackrel{\times}{\simeq}$ TYPICAL GRIP SHOWING THE PIN PRESS-FITTED TWO GRIPS ARE NEEDED. QTY. ORGANIZATION ~ APPROVED: CHECKED: DRAWN: NO. ---2.) TOLERANCES ARE IN MILLIMETERS. (INCHES IN PARENTHESIS) 2.) TOLERANCES ARE AS FOLLOWS: FRACTIONS= ±1.6 (1/16") 2.) TOLERANCES ARE AS FOLLOWS: FRACTIONS= ±1.6 (.03) 2.XXX ± ±1.25 (.01) 2.XXX ± ±1.22 (.005) ANGI FS= ±30' (0.59)15.0 0 REMOVE ALL SHARP EDGES.
SURFACE FINISH FOR MACHINELD SURFACES: 125 UNLESS OTHERWISE SPECIFIED. 15.0 (0.59) 10.0 (0.39) 12.0 (0.47) 15.0(0.59) Π (0.47 <u>+</u> 0.079)<sup>L</sup>  $12.0 \pm 2.0$ NOTES:

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FIG. 2 Loading Pin and Grip Assembly for the Superpave Direct Tension Test

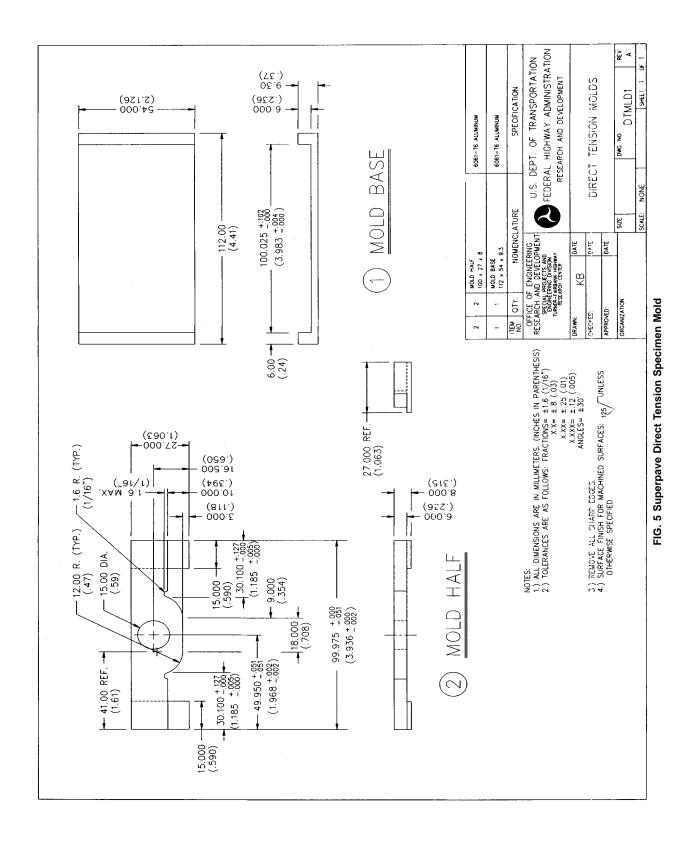


FEDERAL HIGHWAY ADMINISTRATION REV  $\triangleleft$ QF 1 U.S. DEPT. OF TRANSPORTATION HAB H \* RESEARCH AND DEVELOPMENT SPECIFICATION DTINS42 SHEET RING, END DWG. NO. SCALE: NONE NOMENCLATURE SIZE OFFICE OF ENGINEERING RESEARCH AND DEVELOPMENT SPECIA PROLETS AND ENGINEERING DIVISION UNINEERING DIVISION RESEARCH CENTER DATE DATE DATE Ш¥ Ø 10,05 ITEM QTY. 9,95 ORGANIZATION APPROVED: CHECKED: DRAWN: MAY BE MADE FROM 1/4" SCHEDULE 40 ST. ST. TYPE 316 OR 304 2.) TOLERANCES ARE AS FOLLOWS: FRACTIONS=  $\pm 1.6$  (1/16") 2.) TOLERANCES ARE AS FOLLOWS: FRACTIONS=  $\pm 1.6$  (1/16") 2.) X.XE  $\pm 2.8$  (.03) 2.XXXE  $\pm 1.25$  (.01) 2.XXXE  $\pm 1.12$  (.005) ANGLES=  $\pm 30^{\circ}$ Ω Z 3.) REMOVE ALL SHARP EDGES.
4.) SURFACE FINISH FOR MACHINED SURFACES: 125 UNLESS 0THERMSE SPECIFIED. SEAMLESS PIPE MATERIAL: N NOTES: NOTES: 1. MAT Ø 12,700 12,725

FIG. 4 Superpave End Insert Metal Ring for Direct Tension Test

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coefficient of thermal expansion of the asphalt binder and the end tabs is necessary to reduce thermal shrinkage stresses at the interface that otherwise cause bond failures.

6.1.3 Test Chamber for Temperature Control and Testing-The temperature control chamber shall have sufficient space for storing at least eight (8) specimens. The temperature control range for the cooling chamber, without asphalt binder specimens, at all points within the bath, shall be from  $+6^{\circ}$ C to  $-36^{\circ}$ C with temperature stability as a minimum of  $\pm 0.1$  °C. Placing a room temperature specimen in the cold chamber is allowed to cause the chamber temperature between the grips to fluctuate  $\pm 0.2^{\circ}$ C from the target test temperature during testing and isothermal conditioning of the specimen. However, during testing the temperature gradient between the grips shall not exceed  $\pm 0.1$  °C. The temperature measurement shall be accomplished with a calibrated Platinum Resistance Temperature Detector (PRTD) located in the chamber in proximity of test area (NIST Traceable, calibrated at 8 temperatures in the range +6°C to -36°C). If an air based cooling system is used 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. The chamber shall be capable of storing a minimum of eight 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. If an air based cooling system is used, 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  $\pm 0.2$  °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. 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 laser beam.

NOTE 1—The required temperature measurement can be accomplished with an appropriately calibrated platinum resistance thermometer (PRT) or a thermistor. Calibration of the PRT or thermistor can be verified as per 10.1.3. The platinum resistance thermometer meeting DIN Standard 43760 (Class A) is recommended for this purpose.

6.1.4 If fluid is used as a cooling medium, a suitable fluid for temperature control and testing has been found to be an aqueous mixture of 42 % potassium acetate powder and 58 % deionized water by weight. Alcohols have been found to embrittle asphalt binders (that is, affect failure properties in presence of stress) and, therefore, shall not be used as a medium for temperature control and failure testing of asphalt binder specimen.

NOTE 2—Potassium acetate is commercially available in the form of a deicing fluid mixture composed of approximately 50 % potassium acetate and 50 % deionized water. If this commercial fluid mixture is purchased

the required 42 to 58 % potassium acetate to deionized water combination may be obtained by mixing the commercially available 50:50 deicing fluid with deionized or distilled water until a density value between 1.235 and 1.240 is obtained at 25°C. The density may be measured using a suitable hydrometer.

6.1.5 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 Practices E 4. The load and elongation shall be monitored with the data acquisition system such that they can be resolved to 1.0 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 2 N  $\pm$  0.3 N (threshold load) shall be used to mark the point in time where zero reading of the displacement transducer 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 loads typically range from 10 to 250 N depending on the test temperature, grade, aging, and source of the binder. Stress and strain shall be displayed to the nearest 0.1.

6.1.6 *Elongation Measuring and Recording Devices*— Specimen elongation shall be measured between the grips with a displacement transducer. The displacement transducer shall be capable of measuring and controlling grip separation and provide a feedback for elongation rate control with a displacement resolution of 1.0 μm.

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

6.1.6.2 *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 percent strain. 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 aluminum. Molds shall have the dimensions specified in Fig. 4. A release agent along with a silicone-coated release paper, described later in 7.2 and 7.3, shall be used to prevent asphalt binder from adhering to the aluminum mold sides and back plate when making specimen. The release paper is required to prevent asphalt binder from adhering to the bottom plate of the mold.

6.3 *Specimen Storage Plates*—Acrylic plastic, TFE-fluorocarbon or other plastic plates for transferring and storing test specimens in the air cooled environmental chamber. These plates are not required if a fluid cooling bath is used.

6.4 *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 Test Method E 77. An ASTM 133C thermometer as specified in Specification E 1 is suitable. Alternatively, a high precision NIST traceable RTD digital thermometer with an accuracy of better than or equal to  $\pm 0.03^{\circ}$ C is also acceptable for verification of the DTT bath temperature.

6.5 *Freezer*—A freezer capable of maintaining a temperature of  $-15 \pm 5^{\circ}$ C. The freezer is only required when using the air-cooled chamber for testing.

6.6 *Oven*—A forced-air convection oven capable of reaching and maintaining a temperature of  $160 \pm 5^{\circ}$ C for heating asphalt.

# 7. Reagents and Materials

7.1 Fluid for Temperature Control in Systems Equipped with Cooling Baths—An aqueous mixture of 42 % potassium acetate powder and 58 % deionized water by weight shall be used as a fluid for temperature control. Alcohols are not allowed because they have been found to lower the failure properties of all asphalts.

7.2 *Release Agent*—A 50/50 mixture of glycerin and talc is further adjusted as required to obtain a paste of proper consistency. A consistency similar to thin peanut butter was found suitable.

7.3 *Release Paper*—A TFE-fluorocarbon-coated paper coated on both sides with a minimum of 0.3  $\mu$ m of TFE-fluorocarbon has been found to be suitable for use as a release paper. Silicone coated papers may be used as release papers. However, it has been determined that silicone coating lowers the failure properties of certain asphalt binders. These problems typically arise when adequate silicone coating is not present on the release paper.

7.4 *Solvent*—A solvent such as, but not limited to, d-limonene, mineral spirits, or a degreasing spray cleaner formulated for use on asphalt for cleaning molds, end tabs, and plates. A final wipe with an acetone-soaked cloth or paper towel is necessary to ensure a proper bond between asphalt binder and the end-tabs.

7.5 *Cleaning Cloths*—Cloths for wiping molds, end tabs, and plates.

# 8. Hazards

8.1 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. Sampling, Test Specimens, and Test Units

9.1 *Preparing Test Samples*—If unaged binder is to be tested, obtain test samples according to Practice D 140. Set the cooling bath at the desired testing temperature and wait until it stabilizes to within  $\pm 0.1^{\circ}$ C of the desired test temperature. When testing for compliance with Specification D 6373, use the test temperatures specified in Table 1 of Specification D 6373.

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

NOTE 3—Minimum pouring temperatures (readily pours but not overly fluid) are recommended. Heating asphalt binders to temperatures above 165°C should be avoided. However, with some modified asphalts or aged binders, a pouring temperature above 165°C may be required. Temperatures above 180°C should not be used unless absolutely necessary to make the sample sufficiently fluid to pour. In all cases, heating time should be minimized. During the heating process, the sample should be covered and stirred occasionally to ensure homogeneity. Use caution during stirring to avoid trapping air bubbles in the asphalt binder.

9.2 Coat the specimen forming surfaces of the two mold side plates of the aluminum mold with the release agent. The coating shall be applied so as to produce a thin uniform film on the metal surface such that no part of the metal surface is exposed. Place a single precut sheet of release paper on the bottom plate of the mold. Slide one side plate on to the bottom plate over the release paper. Place the end tabs into both ends of the mold. Slide the other side plate on to the bottom plate to complete the assembly of the mold. Place the molds and end tab assembly in a sand bath placed in an oven maintained at the same temperature at which the asphalt binder is heated for no more than  $7 \pm 2$  min. After the required  $7 \pm 2$  min pre-heating time, the sand bath and the molds are removed from the oven for pouring hot asphalt binder in the molds, as explained in paragraph 9.3. If molds are left in the oven for longer than 9 min, it will affect the release properties of the glycerin and talc coating. The sand bath shall be preheated to the required temperature before placing the molds.

9.3 Remove mold and the sand bath from oven and place it on a flat surface. Pour hot asphalt binder into the mold starting from one end of the cavity and moving across the cavity 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. The mold should be overfilled to allow for shrinkage as the specimen cools to ambient temperature.

NOTE 4—Research data suggests that more consistent failure stress results are obtained (coefficient of variation less than 10 %) by heating the assembled molds in a sand bath placed in the oven. An aluminum foil or a piece of cloth should be placed over the sand bath to avoid incorporating sand particles in the mold and the specimen. Only two molds should be heated and poured at a time. Experience suggests that pouring more than two molds at a time will excessively cool the asphalt binder thereby producing specimen of inconsistent geometry.

9.4 After pouring the test specimen, allow the molds to anneal in the sand bath for  $5 \pm 1$  min. Next, remove the molds from the sand bath and allow them to cool on the bench top at ambient temperature for 30–60 min. Do not quench (quick or instantaneous cooling) the specimen to achieve ambient temperature (25°C or lower).

9.5 As soon as the specimen has cooled to room temperature, trim off the excess asphalt binder with an approximately 165°C straight edge (for example, a flat stiff heavy gage putty knife) 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 end tabs and the asphalt binder is not damaged. Trim off the specimen in a consistent manner. Pull the hot knife 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 end tab. Let specimen sit at ambient temperature for 10-15 min after trimming.

# 9.6 Specimen Demolding Procedure:

9.6.1 Prior to demolding, place two side plates of an unused aluminum mold (see Fig. 4) into the cooling bath or air chamber and allow them to cool to the test temperature. These side plates will be used as holders for transferring the specimen to the test bath or chamber.

9.6.2 Prepare a specimen transfer plate as follows: Place an unused aluminum bottom plate of the mold (see Fig. 4) upside down on the work bench so as to form a transfer plate. Place two release papers so that they overlap lengthwise and cover the surface of the transfer plate.

9.6.3 Demold the trimmed specimen by gently sliding the specimen and the two mold side plates towards one edge of the bottom plate until the side plate nearest to the edge is halfway across the edge. Pivot the overhanging side plate downwards using gentle pressure and demold that side.

9.6.4 Replace the just-released plate and now slide the specimen and the two side plates (one side demolded but reassembled) towards the other edge and repeat the procedure given in 9.6.3.

9.6.5 Again, repeat the demolding procedure described in 9.6.3, except this time, remove one side plate completely and gently mount one of the cold side plates from the cooling bath or chamber onto that side of the specimen.

9.6.6 Demold the other mold side plate and mount the other cold side plate from the cooling bath or chamber.

9.6.7 At this point, the specimen and mold assembly should look exactly like the trimmed specimen before demolding except the two side plates are now replaced with cold side plates that were placed in the cooling chamber or bath before the demolding process began.

9.6.8 Now turn the cold specimen mold assembly upside down by holding the mold assembly in the center between two fingers of each hand. Place the upside down mold assembly on the transfer plate so as to form a cross, that is, the mold assembly must be perpedicular to the transfer plate covered with release papers.

9.6.9 Gently remove the bottom plate from the upside down specimen assembly resting on the transfer plate by gently sliding it off. The release paper at the bottom of the specimen is now exposed. Hold the two side plates so as to secure the specimen while gently removing the release paper.

9.6.10 Carry the transfer plate with the upside down but exposed specimen to the test chamber or bath. Immediately place the specimen (along with the two cold side plates) into the cooling bath or chamber onto one of the trays.

9.6.11 Remove the two side plates after a two minute cool-down period. Some softer grades may require more than two minutes before removing the side plates.

9.6.12 Flip the just-released specimen in the test bath or chamber so that the trimmed side is up. The specimen must

always be tested with the trimmed side facing up. The transfer plate covered with the release papers should never be placed into the bath. Measurement of specimen dimensions after demolding is not necessary since dimensional tolerances are closely controlled in the molding process.

NOTE 5—Minimize distortion of the specimen during demolding. The variability in failure properties is increased if distorted (bent along or perpendicular to the loading axis) specimen are tested. A distorted specimen yields lower failure values.

#### 10. Calibration and Standardization

10.1 Verify calibration of the displacement transducer, load cell, and temperature transducer.

NOTE 6—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.

10.1.1 Load Cell and Displacement Transducer Verification-A specially designed standard shall be used to verify the operation of both the load cell and the displacement transducer. The verification standard shall have a spring rate of approximately 135 N/mm and shall be capable of withstanding a 500.00 N load without damage. The verification standard shall be supplied by the manufacturer of the direct tension test system along with the stress-strain characteristics. The manufacturer shall provide a certificate with each verification standard which guarantees the NIST traceability of the equipment used to determine the stress-strain characteristics of the standard. Verify calibration of the load cell and displacement transducer at least every 6 months and when measurements are suspect. Verification of the load cell shall be in accordance with Practices E 4. Verification of the displacement transducer shall be in accordance with Practice E 83.

10.1.2 *Verification of Elongation Rate*—The elongation rate shall be verified using the verification standard discussed in 10.1.1. To verify the elongation rate, elongation shall be plotted as a function of elapsed time. The resulting plot shall be a straight line with a slope 1.00 mm/min.

Note 7—Measurements are typically made at -18  $\pm$  0.1°C for convenience. Allow 10 min for the verification standard to achieve thermal equilibrium.

10.1.3 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 Test Method E 220. Alternatively, a high precision NIST traceable RTD digital thermometer with an accuracy of better than or equal to  $\pm 0.03$  °C is also acceptable for verification of the DTT bath temperature. Place the thermometer or the RTD in the environmentally controlled chamber and hold the RTD in intimate contact with the bulb of the thermometer. When the thermometer and the temperature detection device have reached equilibrium, compare the temperature indicated on the readout for the RTD to the temperature observed by reading the thermometer. If the temperature indicated by the thermal detector does not agree with the mercury in glass thermometer within  $\pm 0.1$  °C, apply an appropriate temperature correction to the temperature measurement indicated by the temperature detector or further calibration or maintenance is required.

# 11. Conditioning

11.1 Condition the specimens in the cooling bath or chamber at the test temperature for  $60 \pm 5$  min. Adhere carefully to time schedule to avoid testing variability that is caused by physical hardening.

# 12. Procedure

12.1 Prepare six test specimens according to Section 9.

12.2 Using tongs, mount a specimen on the load frame by matching the holes on the end tabs at each end of the specimen to the loading pins on the load frame. If the holes do not match with the pins, mount one of the pins with a specimen end and adjust the load frame by moving the thumb wheel on the control box until the other end of the specimen falls into place as shown. Check the seating of the specimen by running the tongs lightly on the end tabs at each end of the specimen.

12.3 Once the specimen is on the loading pins, remove the slack between the specimen and the loading pins.

12.3.1 For instruments with automatic removal of the slack, the test is also automatically started by the software after zeroing the strain. No manual pressing of the start key is required in that case. The test software will automatically monitor the load, zero the strain, and start the test when the load reaches a value of  $2 \pm 0.3$  N.

12.3.2 For instruments with manual slack removal or if the vertical test setup of an air cooled test chamber is used, then remove the slack manually by manipulating the thumb wheel on the control box for fine adjustment. The thumbwheel is moved in the direction so as to produce tension (stretch the specimen). The thumb wheel is moved until a load of  $2 \pm 0.3$  N is shown on the monitor screen.

12.3.2.1 Once this has been accomplished, recheck the seating of the specimen with the tongs to avoid any erroneous load readings.

12.3.2.2 Repeat the above manual loading procedure until a load of 2  $\pm$  0.3 N is again obtained.

12.4 Set the strain rate to 3 %/min (to allow an elongation rate of 1 mm/min) if it is not preset in the software.

12.5 After the specimen fractures, or after the elongation of the specimen reaches 10 %, whichever comes first, stop the test and remove the specimen or pieces.

12.6 The strain at failure is easily identified as the strain at peak load (maximum stress) when the failure is by fracture (that is, 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. Do not continue the test beyond 10 % strain; record the failure strain as "greater than 10 %."

Note 8—If the asphalt binder can be stretched to 10% without fracture, it meets the requirements of Specification D 6373 at the test temperature.

12.7 Record failure load and elongation at failure. If the specimen fails in the throat section, record the data for failure but note that it failed in the throat. If the specimen fails to fracture at 10 % elongation, record the load at 10 % elongation

and the fact that the specimen did not fracture.

NOTE 9—While the acceptable failure is in the gage section (18 mm long constant cross-section portion), ideally failure should occur in the center of each specimen. The location of failure and the consistency or repeatability of failure data is directly related to straightness, how carefully the specimens are prepared, and how well they are mounted prior to testing.

12.8 Repeat 12.2-12.7 for the remaining five specimens.

12.9 After testing is complete, discard the asphalt portions of the spent specimens and clean the end tabs by soaking them in solvent and wiping with a soft cloth. After wiping the end tabs, use a detergent soap solution to remove any oil film residue left by the mineral spirit cleaner. Alternatively, use a degreasing spray cleaner. Clean the end tabs thoroughly. A grease film on the asphalt bonding area can create a weak bond causing bond failures.

## 13. Calculation or Interpretation of Results

13.1 For each test result, compute the failure stress by dividing the failure load by the original area of the test specimen cross-section (6 by 6 mm) as shown in Eq 1:

$$\sigma_f = P_f / A \tag{1}$$

where:

$$\sigma_f$$
 = failure stress, MPa,

$$\vec{P}_f$$
 = failure load, N, and

A' = original area of cross-section, m<sup>2</sup>.

Note 10—For Information only—for specimens used in this test  $A=36\cdot 10^{-6}\ m^2.$ 

13.2 For each test result, compute the failure strain by dividing the elongation at failure by the original gage length, as shown in Eq 2:

$$\epsilon_f = \delta_f / L_e \tag{2}$$

where:

 $f_f$  = failure strain, mm/mm,

 $\delta_f$  = elongation at failure, mm, and

 $\dot{L}_{e}$  = effective gage length, mm.

Note 11—For Information only—for specimens used in this test, the effective gage length,  $L_e$ , has been determined to be 33.8 mm.

13.3 Discard the two test results with the lowest failure stress. If two or more test results have the same failure stress but different failure strains, discard the test result with lower failure strain.

NOTE 12—Research data suggests that failure stress values of asphalt binder follows a Weibull distribution. In a Weibull distribution, high strength values that are from the overall population of the distribution are expected. Research also shows that discarding two lowest values conforms the failure stress data to the Weibull distribution.

13.4 Calculate the mean and standard deviation for failure stress and failure strain for the remaining four test results.

## 14. Report

- 14.1 Report the following information:
- 14.1.1 Sample identification,
- 14.1.2 Date and time of test,
- 14.1.3 Test temperature, to the nearest 0.1°C,
- 14.1.4 Rate of elongation, to the nearest 0.01 mm/min.,

14.1.5 Average failure strain and standard deviation, to the nearest 0.01 percent,

14.1.6 Average failure stress and standard deviation, to the nearest 0.01 MPa,

14.1.7 Peak load, to the nearest N, and

14.1.8 Type of fracture observed (fracture or no fracture, gage or throat).

#### 15. Precision and Bias

15.1 The precision of the procedure for measuring failure stress, failure strain, and failure energy is being determined and will be available on or before March 2002. It is not feasible to specify the precision of the procedure at this time because equipment procurement, required training and experiment design is under way. Therefore, this standard should not be used for acceptance or rejection of material for purchasing purposes.

NOTE 13—*Precision*—The precision of this method is being determined using interlaboratory round robin tests. For your information, until that

data is available, a multi-operator (70 operators) data set was used to determine the repeatability of the test method. An unmodified PG 67-22 asphalt binder was used to generate this data set. This data set was collected and saved during training sessions conducted by a manufacturer for various state agencies and other users. The precision estimates obtained using this data may be considered preliminary, as the requirements of Practice C 670 were not followed in experiment design or data analysis.

Property	Coefficient of	Acceptable Range of
	Variation, %	Coefficient of Variation, %
Failure Stress, MPa	8.5	24.1

15.2 *Bias*—There are no acceptable reference values for the properties determined in this test method so bias for this test method cannot be determined.

## 16. Keywords

16.1 asphalt binder; direct tension; failure; failure strain; fracture; thermal cracking

# APPENDIX

#### (Nonmandatory Information)

## X1. ALTERNATIVE TEST PROCEDURE FOR SYSTEMS WITH FORCED AIR BASED COOLING

X1.1 After  $60 \pm 10$  min, mount the specimen on the pins using the environmental chamber hand access port so that the backface of the end tab 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 end tabs, do not touch the asphalt binder.

NOTE X1.1—Air currents for the circulation fan may cause the end tabs 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 end tab against the face of the grip. It is important that the end tab 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 to 10 mm thick with an outer diameter of approximately 10 mm. The inside diameter of the washer should 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 end tab in place during the test.

X1.2 Select the desired strain rate and load the specimen to failure. Select a strain rate that gives an effective gage section elongation rate of  $1.00 \pm 0.01$  mm/min when testing for compliance with Specification D 6373. If a test specimen fails outside the gage area of the specimen (from throat to throat), discard the test.

X1.3 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 1 to 2 N load; this may reduce testing variability. As soon as the 1 to 2 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 12.4.

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