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# Standard Test Methods for Rubber Property—Brittleness Point of Flexible Polymers and Coated Fabrics[1]

This standard is issued under the fixed designation D 2137; 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 (ε) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 These test methods cover the determination of the lowest temperature at which rubber vulcanizates and rubber-coated fabrics will not exhibit fractures or coating cracks when subjected to specified impact conditions.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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:*

D 751 Test Methods for Coated Fabrics[2]

D 832 Practice for Rubber Conditioning for Low-Temperature Testing[3]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[3]

## 3. Summary of Test Methods

3.1 A specified number of specimens is given a single-impact under specified impact and temperature conditions until the temperature is found at which no failures occur. This is defined as the brittleness temperature.

3.2 There are two test methods.

3.2.1 *Test Method A*— Covers the determination of the lowest temperature at which rubber vulcanizates will not fracture or crack.

3.2.2 *Test Method B*— Covers the determination of the lowest temperature at which rubber-coated fabrics will not fracture or exhibit coating cracks.

3.3 The test can be done either in a liquid heat transfer media or in a gaseous media.

## 4. Significance and Use

4.1 These test methods cover the evaluation of rubber materials or fabrics coated therewith subjected to low-temperature flex with an impact under well-defined conditions of striker speed. The response is largely dependent on effects of low temperatures such as crystallization, incompatibility of plasticizer, or the inherent dynamic behavior of the material itself. Data obtained by these test methods may be used to predict the product behavior in applications where the conditions are similar to those specified in these test methods.

4.2 These test methods have been found useful for specification and development purposes but do not necessarily indicate the lowest temperature at which the material may be used.

## 5. Apparatus

5.1 *Specimen Clamp*, designed so as to hold firmly the specimen(s) as cantilever beams (Fig. 1).

5.2 *Striker*—The edge of the striker shall have a radius of 1.6 ± 0.1 mm (0.063 ± 0.005 in.). The edge shall move relative to the specimen at a rectilinear speed of 2.0 ± 0.2 m/s (6.6 ± 0.6 ft/s) at impact and immediately after. The speed of the solenoid-activated striker should be frequently calibrated by the method described in the annex. Other types of testers shall be calibrated according to their appropriate methods. In order to have the required speed, care must be taken to ensure that the striking energy of at least 3.0 J per specimen is used.

NOTE 1—The striker may be motor-driven, solenoid-operated, gravity-activated or spring-loaded. The motor-driven tester should be equipped with a safety interlock to prevent striker motion when the cover is open.

5.2.1 *Position of striking edge*—The distance between the center line of the striking edge and the clamps shall be 8.0 ± 0.3 mm (0.31 ± 0.01 in). The clearance between the striking arm and the clamp at and immediately following impact shall be:

5.2.1.1 *Test Method A*— 6.4 ± 0.3 mm (0.25 ± 0.01 in)

5.2.1.2 *Test Method B*— Listed as follows:

| Specimen Thickness, mm (in.) | Clearance, mm (in.) |
|---|---|
| 1.65 to 2.20 (0.065 to 0.087) | 6.4 ± 0.3 (0.25 ± 0.01) |
| 1.05 to 1.64 (0.041 to 0.064) | 5.7 ± 0.3 (0.22 ± 0.01) |
| 0.55 to 1.04 (0.022 to 0.040) | 5.2 ± 0.3 (0.20 ± 0.01) |
| 0.10 to 0.54 (0.004 to 0.021) | 4.8 ± 0.3 (0.19 ± 0.01) |

NOTE 2—The dimensional requirements for Test Method B may be

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.14 on Time and Temperature-Dependent Physical Properties.

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[2] *Annual Book of ASTM Standards*, Vol 09.02.

[3] *Annual Book of ASTM Standards*, Vol 09.01.

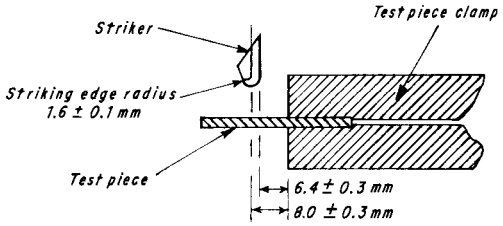



**FIG. 1 Specimen Clamp and Striker**

obtained by fabricating individual plates to fit the specimen holder illustrated in Fig. 1.

5.3 *Tank or Test Chamber*—A tank for liquid heat transfer media or a test chamber for gaseous media is required. To ensure thorough circulation of the heat transfer medium, a stirrer should be provided for liquids and a fan or blower for gaseous media.

5.4 *Heat Transfer Media*:

5.4.1 *Liquid Heat Transfer Medium*—Methanol is the recommended heat transfer medium. Since methanol is flammable and toxic, the bath should be isolated in a closed hood.

NOTE 3—Any other liquid heat transfer medium that remains fluid at the test temperature and will not appreciably affect the material tested may be used. The following materials have been used down to the indicated temperatures.

| | °C |
|---|---|
| Dow Corning—200 fluids: | |
| 5 $mm^2/s$ viscosity | −60 |
| 2 $mm^2/s$ viscosity | −76 |
| Methanol | −90 |
| Propyl Alcohol | −120 |

NOTE 4—The desired temperature may also be obtained by filling the tank with the heat transfer medium and lowering its temperature by the addition of liquid carbon dioxide controlled by a solenoid-activated unit with an associated temperature control. Where temperatures below that obtainable by solid or liquid carbon dioxide are required, liquid nitrogen may be used.

5.4.2 *Gaseous Medium*— A gaseous medium may be used provided ample time is allowed for the specimens to reach temperature equilibrium with the temperature of the medium.

NOTE 5—The apparatus may be used in a gaseous medium if it can be shown that low temperature will not affect the operation of the solenoid-activated impact mechanism.

5.5 *Temperature Control*—Suitable means shall be provided for controlling the temperature of the heat transfer medium within ±0.5°C (±1°F) if the medium is liquid and within ±1°C (±1.8°F) with gaseous medium.

5.5.1 Temperature monitoring is done with a thermocouple or other temperature-sensing device with associated temperature indicator graduated in 0.5°C (1°F) divisions and having a range suitable for the temperatures at which the tests are to be made. The thermocouple is preferably constructed of copper-constantan wire having a diameter between 0.2 and 0.5 mm (32 to 24 Awg) and shall be fusion-bonded at the junction. It shall be located as near the specimens as possible. A thermometer may also be used if it can be shown to agree with the thermocouple and other devices that respond rapidly to temperature change.

5.5.2 Automatic changes in temperature of a liquid medium may be obtained by means of a system consisting of an externally cooled tank connected to the test area with suitable tubing, a thermoregulator, a pump, an electric immersion heater, and mercury switches. The regulator, alternately activating both the pump and heater through the mercury switches, controls the amount of liquid coolant being pumped to the test area as well as the amount of heat coming from the heater.

5.5.3 Manual temperature changes for liquid media may be accomplished with powdered carbon dioxide (dry ice) and an electric immersion heater.

## 6. Time Lapse Between Vulcanization and Testing

6.1 For all test purposes, the minimum time between vulcanization and testing shall be 16 h.

6.2 For nonproduct tests, the maximum time between vulcanization and testing should be four weeks, and for evaluation intended to be comparable, the tests should be carried out after the same time interval.

6.3 For product tests, whenever possible, the time between vulcanization and testing should not exceed three months. In other cases, tests should be made within two months of the date of receipt by the customer.

## 7. Test Specimens

7.1 *Test Method A*— The die-punched Type B specimens as illustrated in Fig. 2 shall be used.

NOTE 6—Type A strips 40 ± 6 mm (1.6 ± 0.25 in.) long, 6 ± 0.5 mm (0.25 ± 0.02 in.) wide, and 2.0 ± 0.2 mm (0.08 ± 0.01 in.) thick (see Fig. 3) may be used but will not necessarily give comparable results.

NOTE 7—Specimens of other than 2.0 ± 0.2 mm (0.08 ± 0.01 in.) thicknesses may be used provided it can be shown that they give equivalent results for the material being tested.

7.2 *Test Method B*— Type A specimens shall be used. They should be die-punched with the longer dimensions parallel to the lengthwise direction of the coated fabric, unless otherwise specified, and be 40 ± 6 mm (1.6 ± 0.25 in.) long and 6 ± 0.5 mm (0.25 ± 0.02 in.) wide.

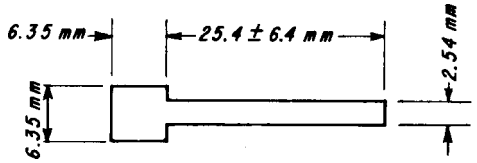
NOTE 8—Sharp dies must be used in the preparation of specimens if reliable results are to be achieved. Careful maintenance of die cutting edges is extremely important and can be obtained by frequent light honing touching up with jeweler's honing stones.

## 8. Conditioning

8.1 *Test Method A*— Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for no less than 16 h prior to testing.

8.2 *Test Method B*— The test specimens shall be conditioned prior to the test in accordance with the standard conditions in Test Methods D 751.

8.3 Where long-time effects, such as crystallization, incompatibility, etc., of the material, are to be studied, the test




NOTE 1—The test piece thickness is 2.0 ± 0.2 mm.

**FIG. 2 Modified T-50 Test Piece**

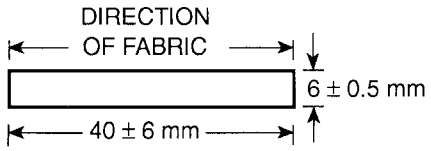



**FIG. 3 a Type A Specimen**

specimens may be conditioned in accordance with Practice D 832.

## 9. Procedure

9.1 *Test Method A*:

9.1.1 *Test with Liquid Heat Transfer Medium*:

9.1.1.1 Prepare and bring the bath to a temperature below the expected lowest temperature of non-failure. Place sufficient liquid in the tank to ensure approximately 25 mm (1 in.) liquid covering the test specimens.

9.1.1.2 Mount five Type B specimens in the apparatus with the entire tab in the clamp. Immerse the specimens for 3.0 ± 0.5 min at the test temperature.

NOTE 9—If five Type A specimens are used, a minimum of 6 mm (0.25 in.) of the specimen length must be held in the clamp.

NOTE 10—If the energy capacity of the apparatus causes the speed of the striker to fall below 1.8 m/s (6 ft/s), a smaller number of specimens may be mounted.

9.1.1.3 After immersion for the specified time, record the actual test temperature and deliver a single impact to the specimens.

9.1.1.4 Examine each specimen to determine whether or not it has failed. Failure is defined as any crack, fissure, or hole visible to the naked eye, or complete separation into two or more pieces. When a specimen has not completely separated, bend it to an angle of 90° in the same direction as the bend caused by the impact, then examine it for cracks at the bend.

9.1.1.5 Repeat the test at the next higher temperatures at 10°C intervals using new specimens each time until no failure is obtained. Then decrease the bath temperature at 2° intervals. Test at each temperature to determine the lowest temperature at which no failures occur. Record this temperature as the lowest temperature of non-failure.

9.2 *Test with Gaseous Heat Transfer Medium*:

9.2.1 Adjust the refrigerating unit and bring the test chamber, test apparatus, and specimens to thermal equilibrium at the desired temperature (see Note 4). An alternative method is to place the striker and specimen clamp through the top of the refrigerating unit with the solenoid remaining outside the unit and insulated from the cold air.

9.2.2 The actual testing is performed in the same manner as described in 9.1.1.

9.3 *Testing of Materials from Approved Supplier*:

9.3.1 For inspection and acceptance of materials received from an approved supplier, it shall be satisfactory to accept lots on the basis of testing ten specimens (five at a time) at a specified temperature as stated in the relevant material specifications. Not more than five shall fail. Should there be no failures in the testing of the first five specimens, the testing of the second five specimens is not required.

9.3.2 It shall be satisfactory to accept rubber compositions on a basis of testing five specimens at a specified temperature, as stated in the relevant material specification. None shall fail.

9.4 *Test Method B*:

9.4.1 Follow the instructions in accordance with 9.1.1.1 through 9.1.1.3, except that Type A specimens shall be used. The specimens shall be examined for any visible fracture or crack in the coating under a 5× magnifier, after having bent the specimens to an angle of 180° in the same direction caused by the impact.

9.4.2 Use new specimens for each test.

9.4.3 For routine testing of all coated fabrics, subject five specimens to the impact test at a specified temperature as stated in the relevant material specification. None shall fail.

## 10. Report

10.1 Report the following information:

10.1.1 Complete identification of the material tested, including type, source, manufacturer's code designation, form, and date produced, if applicable,

10.1.2 Thickness and type of specimen,

10.1.3 Number of specimens tested at a single impact if other than five,

10.1.4 Conditioning period, method, and procedure,

10.1.5 Test method used,

10.1.6 Heat transfer medium used, and

10.1.7 Brittleness temperature to nearest 1°C (2°F).

## 11. Precision and Bias [4]

11.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical calculation details.

11.2 A Type 1 (interlaboratory) precision was evaluated in 1987. Both repeatability and reproducibility are short term. A period of a few days separates replicate test results. A test result, as specified by this test method, is obtained on one determination or measurement of the property or parameter in question.

11.3 Four different materials were used in the interlaboratory program. These were tested in seven laboratories on two different days.

11.4 The results of the precision calculations for repeatability and reproducibility are given in Table 1, in ascending order of material average or level, for each of the materials evaluated. Measurements, in° C, have been transformed to kelvin as the brittleness temperature of one of the materials is approximately 0°C.

11.5 *Repeatability, r*, does not vary over the range of material levels as evaluated. Reproducibility varies over the range of material levels evaluated.

11.6 The precision of these test methods may be expressed in the format of the following statements which use an "appropriate value" of *r*, *R*, (*r*), or (*R*), that is, that value to be used in decisions about test results (obtained with the test method). The *appropriate value* is that value of *r* or *R* associated with a mean level in Table 1 closest to the mean level under consideration at any given time, for any given material, in routine testing operations.

[4] Supporting data are available from ASTM Headquarters. Request RR: D11-1052.

**TABLE 1 Type 1 Precision of Brittleness Temperature (K)**

NOTE 1—The precision data are calculated in kelvin to avoid large values of (*r*) and (*R*) as Material B approximates 0°C in its measurement or brittleness temperature.

| Material | Average, K(°C) | Within Laboratory[A] | | | Between Laboratory[A] | | |
|---|---|---|---|---|---|---|---|
| | | $S_r$ | *r* | (*r*) | $S_R$ | *R* | (*R*) |
| D, IR | 217.6 (−55.5) | 0.00 | 0.00 | 0.0 | 1.65 | 4.67 | 2.1 |
| C, NR | 219.5 (−53.6) | 0.75 | 2.13 | 0.9 | 2.49 | 7.06 | 3.2 |
| A, SBR | 227.6 (−45.5) | 0.96 | 2.72 | 1.1 | 4.18 | 11.84 | 5.2 |
| B, NBR | 260.0 (−13.1) | 0.75 | 2.13 | 0.8 | 3.71 | 10.52 | 4.0 |
| Pooled values[B] | 230.1 (−43.0) | 0.73 | 2.07 | 0.9 | 3.28 | 9.28 | 4.0 |

[A] $S_r$ = repeatability standard deviation.
*r* = repeatability = 2.83 times the square root of the repeatability variance.
(*r*) = repeatability (as percent of material average).
$S_R$ = reproducibility standard deviation.
*R* = reproducibility = 2.83 times the square root of the reproducibility variance.
( *R*) = reproducibility (as percent of material average).
[B] No values ommitted.

11.7 *Repeatability*—The repeatability, *r*, of these test methods have been established as the *appropriate value* tabulated in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

11.8 *Reproducibility*— The reproducibility, *R*, of these test methods have been established as the *appropriate value* tabulated in Table 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

11.9 Repeatability and reproducibility expressed as a percent of the mean level, (*r*) and ( *R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percent of the arithmetic mean of the two test results.

11.10 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by these test methods. Bias, therefore, cannot be determined.

## 12. Keywords

12.1 brittleness; flexibility; low temperature; rubber-coated fabrics

## ANNEXES

**(Mandatory Information)**

### A1. SPEED CALIBRATION OF THE SOLENOID-ACTUATED BRITTLENESS TESTER PRIOR TO ACTUAL TESTING

A1.1 Calibration is accomplished by measuring the height, *h*, to which a steel ball, suspended on the striker mechanism of the tester, rises after the striker has had its upward motion halted by contact with a mechanical stop. The ball is accelerated in such a manner that the law governing a freely falling body applies. The velocity, *v*, of the striker is readily calculated from the following expression:

$$v = \sqrt{2gh} \quad \text{(A1.1)}$$

A1.2 — *Securing Ball Support*—Remove either one of the nuts that fasten the striking bar guide rods to the solenoid armature yoke. Place the small hole of the ball support (Fig. A1.1) over the guide rod and replace and secure the nut.

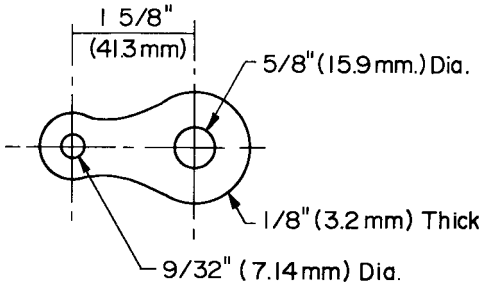


**FIG. A1.1 Ball Support**

A1.3 *Adjusting Stroke of Striker*—Remove the metal guard from around the solenoid. Spread open the rubber bumper (Fig. A1.2) and insert it around the armature. Replace the solenoid guard. Insert a typical rubber or plastic specimen into the specimen holder of the tester. Raise the striking mechanism by hand until the end of the stroke is reached. It is essential that, with the striking mechanism raised to its maximum height, the striker bar of the tester be in contact with the specimen but that the bar not be in the plane of the specimen. If the striker bar is not in contact with the specimen, the rubber bumper must be removed and replaced by a thinner bumper. Conversely, if the striker bar moves into the plane of the specimen, the bumper

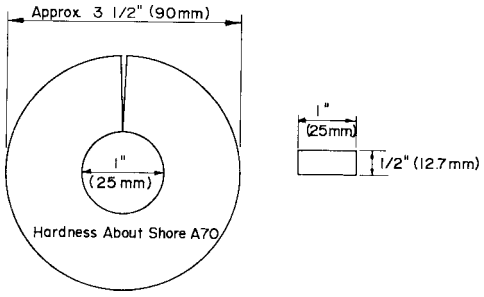



**FIG. A1.2 Rubber Bumper**

must be replaced by a thicker one.

A1.4 *Placement of Ball and Measuring Tube*—Place a 19-mm (¾-in.) diameter steel ball on the ball holder. (In theory, the upward flight of the ball is independent of the mass of the ball. However, if the mass is too large, the motion of the striker bar may be impeded.) Clamp a glass or clear plastic tube with a minimum inside diameter of 25.4 mm (1 in.) in a vertical position directly over the ball. The tube should contain a scale divided into 5-mm (¼-in.) intervals. The zero position on the scale should be aligned with the top of the ball when the ball is at the top of the stroke of the striker mechanism.

A1.5 *Measurement and Calculation*—With the tester equipped as described above and devoid of test specimens and immersion medium, fire the solenoid and read the ball height to the closest 5 mm (¼ in.). Make at least five measurements. Average all results and convert the average to metres (or feet). Determine the striker speed, $v$, from the following equation:

$$v = \sqrt{2gh} \quad \text{(A1.2)}$$

where:
$v$ = speed, m/s (or ft/s).
$g$ = 9.8 m/s$^2$ (32.2 ft/s$^2$), and
$h$ = average ball height, m (or ft).

NOTE A1.1—Calibration measurements should be made with the tester supported on a non-resilient surface, such as a laboratory bench or concrete floor. Resilient mountings tend to absorb some of the striker energy causing low ball height values.

## A2. SPEED CALIBRATION OF THE SOLENOID-ACTUATED BRITTLENESS TESTER DURING ACTUAL TESTING

A2.1 With the tester equipped with ball support, ball, and measuring tube (see Annex A1), but without the rubber bumper (tester in the normal operating condition) and devoid of test specimens and immersion medium, fire the solenoid and read the ball height to the closest 5 mm (¼in.). Make ten measurements. From the lowest and highest ball height readings, determine the range in striker speed, using Eq A1.1. This range is termed "range of speed at the top of the stroke."

A2.2 With the tester equipped in accordance with A2.1, but also with test specimen(s) and immersion medium, conduct the brittleness test in accordance with Section 9. Read the ball height each time the solenoid is fired. Convert the ball height to speed as shown in A1.5. If the speed lies within the predetermined range of speed at the top of the stroke, the test will be considered valid. If the speed lies outside of the predetermined range, the test will be invalid and should not be reported. Should successive tests be invalid, adjustments should be made to bring the speed at the top of the stroke within the acceptable, predetermined range. This may be accomplished by reducing the number of specimens tested per impact or by changing from Type A to Type B specimens.

A2.3 The following example typifies the entire speed calibration procedure for solenoid actuated testers:

A2.3.1 Using the procedure of Annex A1, the striker speed at point of impact of a tester devoid of test specimens and immersion medium was found to be 1.9 m/s (6.2 ft/s). This speed is within the specified limits of 5.2.

A2.3.2 Using the procedure of A2.1, with the tester devoid of test specimens and immersion medium, the range of striker speed at the top of the stroke was found to be 2.5 to 2.7 m/s (8.2 to 8.7 ft/s). This range becomes the acceptable range for this series of tests. The acceptable range should be established each time the striker speed at point of impact is determined (see Annex A1).

A2.3.3 Using the procedure of A2.2, with the tester containing a test specimen(s) and immersion medium, the speed at the top of the stroke during the first solenoid firing was found to be 2.5 m/s (8.2 ft/s). This speed was within the acceptable range and the test was valid.

A2.3.4 The speeds at the top of the stroke during the second and third solenoid firings were found to be 2.4 and 2.3 m/s (7.9 and 7.5 ft/s), respectively. These speeds are outside of the acceptable range and both tests are invalid.

A2.3.5 Adjustments were made to increase the speed at the top of the stroke, using the procedures given in A2.2.

A2.3.6 The speeds at the top of the stroke during the fourth and all subsequent solenoid firings were found to lie between 2.5 and 2.7 m/s (8.2 to 8.7 ft/s). The results of all these tests were valid.