Designation: ~~D746 – 07~~ D746 – 13

Standard Test Method for Brittleness Temperature of Plastics and Elastomers by Impact[1]

This standard is issued under the fixed designation D746; 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 This test method covers the determination of the temperature at which plastics and elastomers (as defined by Terminology D883) exhibit brittle failure under specified impact conditions. Two routine inspection and acceptance procedures are also provided.

NOTE 1—When testing rubbers for impact brittleness use Test ~~Methods~~Method D2137. When testing plastic sheeting for impact brittleness, use Test Method D1790.

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

1.3 Due to the potential safety and environmental hazards associated with mercury-filled thermometers, the use of alternative temperature measuring devices (such as thermocouples and RTDs) is encouraged.

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.*

NOTE 2—This test method and ISO 974 ~~(E) are technically equivalent when using the Type B fixture and the Type III specimen, however, the minimum number of specimens that are required to be tested is significantly different when using this test method. The ISO method requires that a minimum of 100 specimens be tested.~~ address the same subject matter, but differ in technical content.

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D618 Practice for Conditioning Plastics for Testing
D832 Practice for Rubber Conditioning For Low Temperature Testing
D883 Terminology Relating to Plastics
D1790 Test Method for Brittleness Temperature of Plastic Sheeting by Impact
D2137 Test Methods for Rubber Property—Brittleness Point of Flexible Polymers and Coated Fabrics
E1 Specification for ASTM Liquid-in-Glass Thermometers
E77 Test Method for Inspection and Verification of Thermometers
E608/E608M Specification for Mineral-Insulated, Metal-Sheathed Base Metal Thermocouples
E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

2.2 *ISO Standard:*
ISO 974 ~~(E)~~ Plastics—Determination of the Brittleness Temperature by Impact[3]

2.3 *ASTM Adjuncts:*
Detailed Drawing of a Typical Clamp[4]

3. Terminology

3.1 *General*—The definitions of plastics used in this test method are in accordance with Test Method D883 unless otherwise specified.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties.30.07).
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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] *ISO Standards Handbook 21*, Vol 1. ISO Standards are available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

[4] A detailed drawing of a typical clamp may be obtained from ASTM Headquarters. Order Adjunct : ADJD0746.

***A Summary of Changes section appears at the end of this standard**

3.2 *brittleness temperature*—that temperature, estimated statistically, at which 50 % of the specimens would probably fail.

3.3 *failed specimen*—the division of a specimen into two or more completely separated pieces or as any crack in the specimen which is visible to the unaided eye.

4. Summary of Test Method

4.1 To determine the brittleness temperature, specimens are secured to a specimen holder ~~with~~using a torque wrench. The specimen holder is immersed in a bath containing a heat-transfer ~~medium that is~~ medium, which has been cooled. The specimens are struck at a specified linear speed and then ~~examined.~~ examined for failures. The brittleness temperature is defined as the temperature at which 50 % of the specimens ~~fail.~~ would probably fail.

5. Significance and Use

5.1 This test method establishes the temperature at which 50 % of the specimens tested would probably fail when subjected to the conditions specified herein. The test provides for the evaluation of long-time effects such as crystallization, or those effects that are introduced by low-temperature incompatibility of plasticizers in the material under test. Plastics and elastomers are used in many applications requiring low-temperature flexing with or without impact. Use data obtained by this method to predict the behavior of plastic and elastomeric materials at low temperatures only in applications in which the conditions of deformation are similar to those specified in this test method. This test method has been found useful for specification purposes, but does not necessarily measure the lowest temperature at which the material is suitable for use.

6. Apparatus

~~6.1 *Type A:*~~

~~6.1.1 *Specimen Clamp and Striking Member*—Design the specimen clamp to hold the specimen or specimens as a cantilever beam. Each individual specimen shall be firmly and securely held in a separate clamp. The striking edge shall move relative to the specimens at a linear speed of 2000 ± 200 mm/s at impact and during at least the following 6.4 mm of travel. In order to maintain this speed on some instruments, it is necessary to reduce the number of specimens tested at one time. The distance between the center line of the striking edge and the clamp shall be 7.87 ± 0.25 mm at impact. The striking edge shall have a radius of 1.6 ± 0.1 mm. The striking arm and specimen clamp shall have a clearance of 6.35 ± 0.25 mm at and immediately following impact. These dimensional requirements are illustrated in Fig. 1. Fig. 2 shows a typical clamp.[4] Use free-fitting clamping screws, 10-32 National Fine Thread.~~

6.1 ~~*Type B:*~~ *Specimen Clamp and Striking Edge:*

6.1.1 *Type A:*

6.1.1.1 Design the specimen clamp to hold the specimen or specimens as a cantilever beam. Each individual specimen shall be firmly and securely held in a separate clamp. The striking edge shall be hardened steel, have a radius of 1.6 ± 0.1 mm and shall move relative to the specimens at a linear speed of 2000 ± 200 mm/s at impact and during at least the following 6.4 mm of travel. In order to maintain this speed on some instruments, it is necessary to reduce the number of specimens tested at one time. The distance between the center line of the striking edge and the clamp shall be 7.87 ± 0.25 mm at impact. The striking edge and specimen clamp shall have a clearance of 6.35 ± 0.25 mm at and immediately following impact. These dimensional requirements are illustrated in Fig. 1. Fig. 2 shows a typical clamp.[4] Use free-fitting clamping screws, 10-32 National Fine Thread.

6.1.2 ~~*Specimen Clamp and Striking Member—Type B:*~~ ~~Design the specimen clamp to hold the specimen or specimens as a cantilever beam. Each individual specimen shall be firmly and securely held in a separate clamp. The striking edge shall move relative to the specimens at a linear speed of 2000 ± 200 mm/s at impact and during at least the following 5.0 mm of travel. In order to maintain this speed on some instruments, it is necessary to reduce the number of specimens tested at one time. The radius of the lower jaw of the clamp shall be 4.0 ± 0.1 mm. The striking edge shall have a radius of 1.6 ± 0.1 mm. The striking edge and specimen clamp shall have a clearance of 3.6 ± 0.1 mm at and immediately following impact. The clearance between the~~

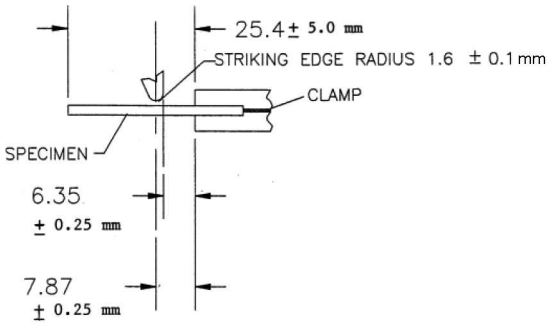


FIG. 1 Dimensional Requirements Between Specimen Clamp and Striking Edge (Type A)

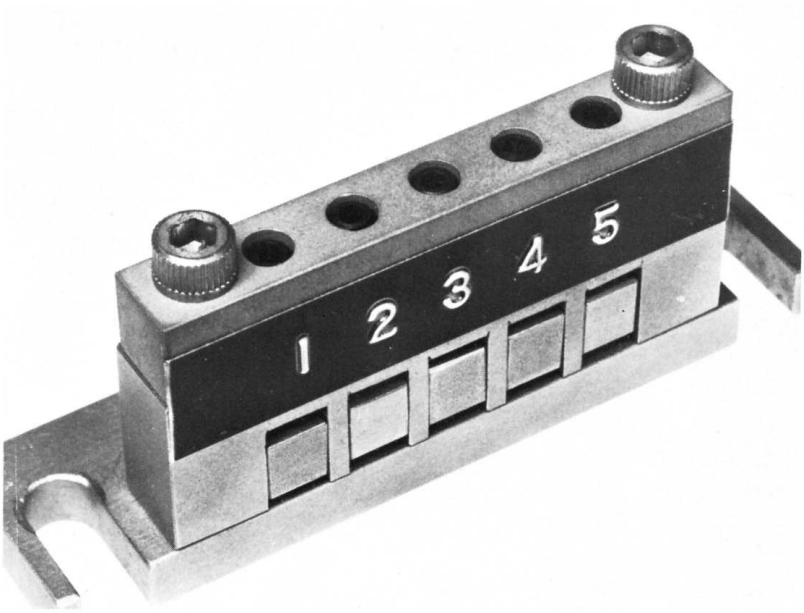
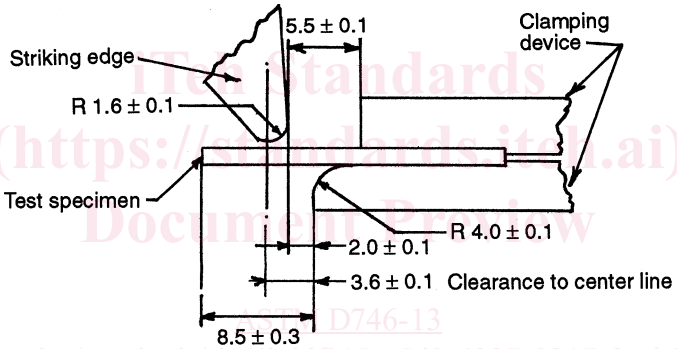


FIG. 2 Typical Clamp (Type A)



NOTE 1—Dimensions are in millimetres.

FIG. 3 Dimensional Details of Striking Edge and Clamping Device, Type B (Positioning of Unnotched Test Specimen)

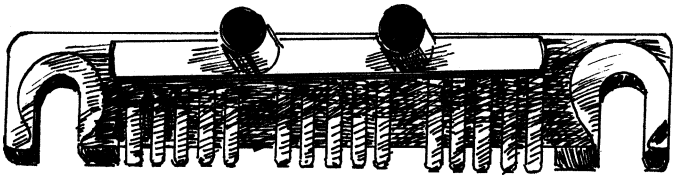


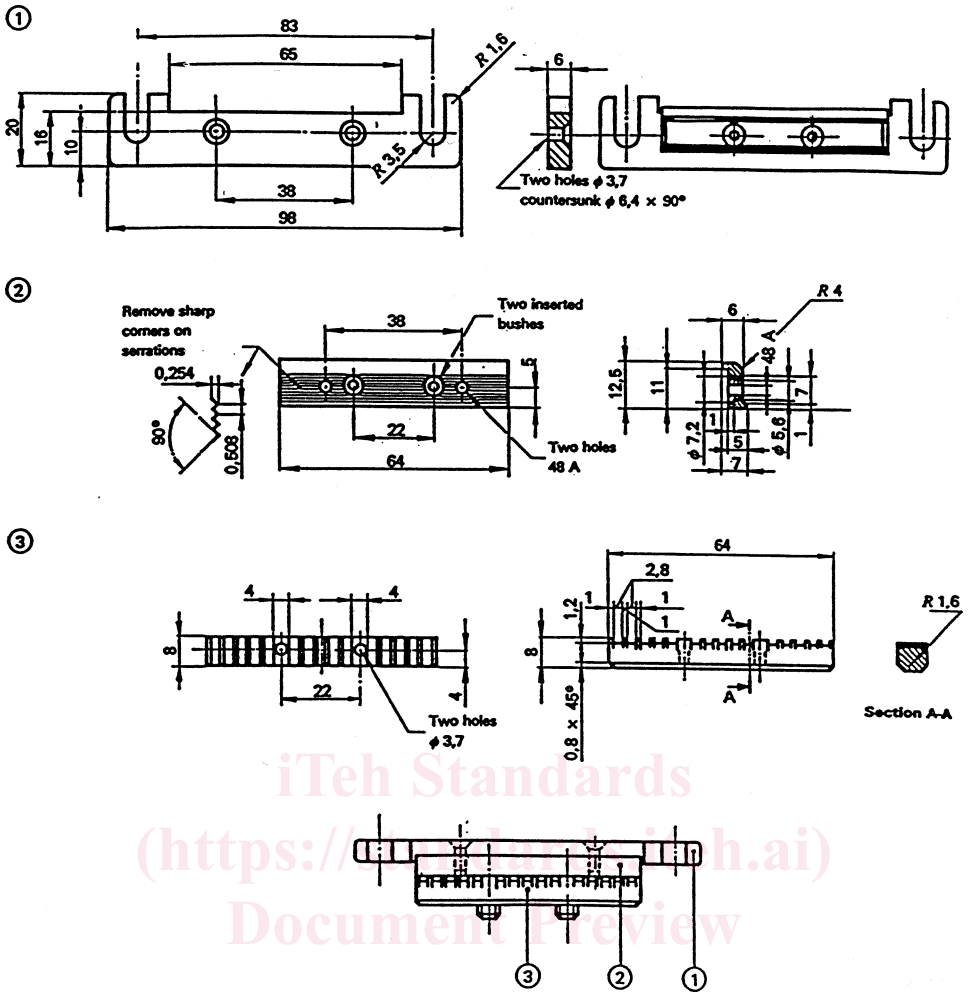
FIG. 4 Assembled Clamp with Test Specimens, Type B

~~outside of the striking edge and the clamp shall be 2.0 ± 0.1 mm at impact. These dimensional requirements of the striking edge and clamping device are illustrated in Fig. 3. Fig. 4 shows a typical clamp. Details of the specimen clamp are given in Fig. 5.~~

6.1.2.1 Design the specimen clamp to hold the specimen or specimens as a cantilever beam. Each individual specimen shall be firmly and securely held in a separate clamp. The striking edge shall be hardened steel, have a radius of 1.6 ± 0.1 mm, and shall move relative to the specimens at a linear speed of 2000 ± 200 mm/s at impact and during at least the following 5.0 mm of travel. In order to maintain this speed on some instruments, it is necessary to reduce the number of specimens tested at one time. The radius of the lower jaw of the clamp shall be 4.0 ± 0.1 mm. The striking edge and specimen clamp shall have a clearance of 3.6 ± 0.1 mm at and immediately following impact. The clearance between the outside of the striking edge and the clamp shall be 2.0 ± 0.1 mm at impact. These dimensional requirements of the striking edge and clamping device are illustrated in Fig. 3. Fig. 4 shows a typical clamp. Details of the specimen clamp are given in Fig. 5.

6.2 *Torque Wrench,* 0 to 8.5 N · m.

NOTE 3—Because of the difference in geometry of the specimen clamps, test results obtained when using the Type A specimen clamp and striking



NOTE 1—Dimensions are in millimetres.

FIG. 5 Details of One Form of Clamp Meeting the Requirements of ~~6.2~~6.1.2

member may not correlate with those results obtained when using the Type B apparatus.

6.3 *Temperature-Measurement System*—The temperature of the heat-transfer medium shall be determined with a temperature measuring device (for example, thermocouple, resistance thermometer, or liquid-in-glass thermometer) having a suitable range for the temperatures at which the determinations are to be made. The temperature-measuring device and the related readout equipment shall be accurate to at least ±0.5°C. The temperature-measuring device shall be located as close to the specimens as possible. Thermocouples shall conform to the requirements of Specification E608/E608M. Resistance temperature devices shall comply with the requirements of Specification E1137/E1137M. Liquid-in-glass thermometers, are described in Specification E1. Use the thermometer appropriate for the temperature range and accuracy required, and calibrate it for the appropriate immersion depth in accordance with Test Method E77.

6.4 *Heat-Transfer Medium*— Use any liquid heat transfer medium that remains fluid at the test temperature and does not appreciably affect the material being tested. Measurement of selected physical properties prior to and after 15-min exposure at the highest temperature used will provide an indication of the inertness of a plastic to the heat transfer medium.

6.4.1 Where a flammable or toxic solvent is used as the cooling medium, follow customary precautions when handling such materials. Methanol is the recommended heat transfer medium for rubber.

NOTE 4—The following materials have been found suitable for use at the indicated temperatures. When silicone oil is used, moisture from the air will condense on the surface of the oil, causing slush to form. If slush collects on the temperature-measuring device as ice, it will affect temperature measurement. When this occurs, remove the ice from the temperature-measuring device.

Material	Temperature, °C
5-mm^2/s viscosity silicone oil	−60
2-mm^2/s viscosity silicone oil	−76
Methyl alcohol	−90

6.5 *Temperature Control*—Suitable means (automatic or manual) shall be provided for controlling the temperature of the heat-transfer medium to within ±0.5°C of the desired value. Powdered solid carbon dioxide (dry ice) and liquid nitrogen are recommended for lowering the temperature, and an electric immersion heater for raising the temperature.

6.6 *Tank,* insulated.

6.7 *Stirrer,* to provide thorough circulation of the heat transfer medium.

NOTE 5—Suitable apparatus is commercially available from several suppliers. The striking member may be motor-driven, solenoid-operated, gravity-actuated, or spring-loaded. Equip the motor-driven tester with a safety interlock to prevent striker arm motion when the cover is open.

7. Test Specimen

7.1 *Type I (for ~~Fixture~~ Type ~~A):~~ A Apparatus):*

7.1.1 *Geometry*—This type of specimen shall be 6.35 ± 0.51 mm wide by 31.75 ± 6.35 mm long as illustrated in Fig. 6.

7.1.2 *Preparation*—Specimens shall be 1.91 ± 0.13 mm thick. Specimens shall be die-punched, cut by hand using a razor blade or other sharp tool, or cut by an automatic machine from flat sheet, or prepared by injection molding.

7.2 *Type II (for ~~Fixture~~ Type ~~A):~~ A Apparatus):*

7.2.1 *Geometry (Modified T-50 Specimen)*—This type of specimen shall be T-shaped, as illustrated in Fig. 6. When using this type of specimen, clamp it so that the entire tab is inside the jaws for a minimum distance of 3.18 mm.

7.2.2 *Preparation*— Specimens shall be 1.91 ± 0.13 mm thick. Specimens shall be die-punched, cut by hand using a razor blade or other sharp tool, or cut by an automatic machine from flat sheet, or prepared by injection molding.

7.3 *Type III (for ~~Fixture~~ Type ~~B):~~ B Apparatus):*

7.3.1 *Geometry*—This type of specimen shall be 20.0 ± 0.25 mm long by 2.5 ± 0.05 mm wide and 1.6 ± 0.1 mm thick as illustrated in Fig. 6.

7.3.2 *Preparation*—Specimens shall be die-punched, cut by hand using a razor blade or other sharp tool, or cut by an automatic machine from flat sheet, or prepared by injection molding.

7.4 Test results will vary according to molding conditions and methods of specimen preparation. It is essential that preparation methods produce uniform specimens. The preferred method of preparation is to use an automatic cutting machine, however specimens that are punched using an arbor press or hydraulically operated press are also acceptable. No matter which preparation method is employed, the specimen edges shall be free of all flash. Specimens that are damaged in any way shall be discarded. If specimens are to be die punched, sharp dies must be used in the preparation of specimens for this test if reliable results are to be achieved. Careful maintenance of die cutting edges is of extreme importance and is obtained by daily lightly honing and touching up the cutting edges with jewelers' hard Arkansas honing stones. The condition of the die is judged by investigating the rupture point on any series of broken specimens. When broken specimens are removed from the clamps of the testing machine it is advantageous to pile these specimens and note if there is any tendency to break at or near the same portion of each specimen. Rupture points consistently at the same place are the indication that the die is dull, nicked, or bent at that particular position, or that some other defect is present.

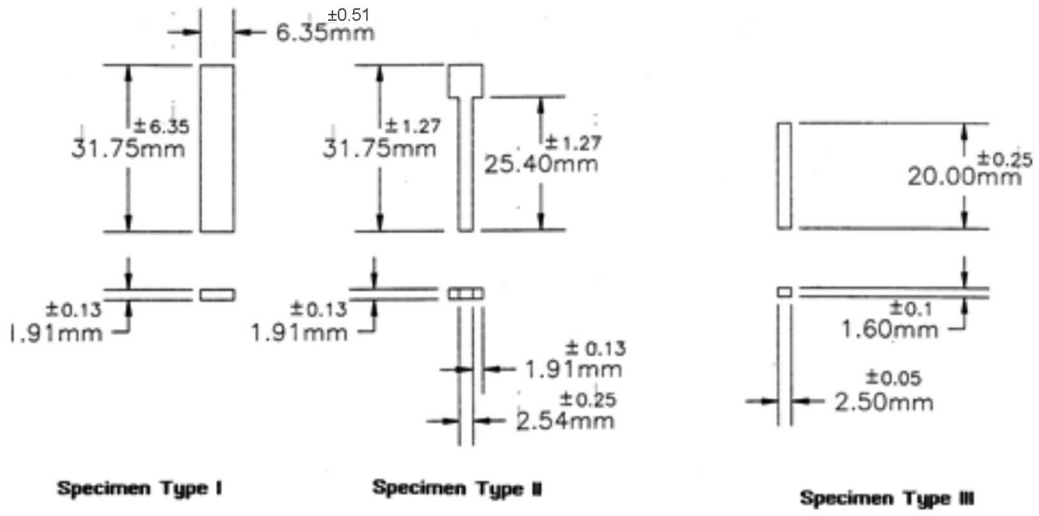


FIG. 6 Specimen Geometry

8. Conditioning

8.1 *Conditioning*— Condition the test specimens at 23 ± 2°C and ~~50 ± 5 %~~50 ± 10 % relative humidity for not less than 40 h prior to the test in accordance with Procedure A of Practice D618 for those tests where conditioning is required. In cases of disagreement, the tolerances shall be ±1°C and ~~±2 %~~±5 % relative humidity.

8.2 Where long-time effects such as crystallization, incompatibility, and so forth, of materials are to be studied, condition the test specimens in accordance with Practice D832.

9. Procedure

9.1 ~~In establishing the brittleness temperature of a material, it is recommended that the test be started at a temperature at which 50 % failure is expected. Test a minimum of ten specimens at this temperature. If all of the specimens fail, increase the temperature of the bath by 10°C and repeat the test using new specimens. If none of the specimens fail, decrease the bath temperature by 10°C and repeat the test using new specimens. If the approximate brittleness temperature is not known, select the start temperature arbitrarily.~~*Routine Inspection and Acceptance:*

9.1.1 For routine inspection of materials received from an approved supplier, it shall be satisfactory to accept lots on the basis of testing a minimum of ten specimens at a specified temperature as stated in the relevant material specifications. Not more than five shall fail.

9.1.2 It shall be satisfactory to accept elastomeric composition on a basis of testing five specimens at a specified temperature, as stated in the relevant material specifications. None shall fail.

~~9.2 Prior to beginning a test, prepare the bath and bring the apparatus to the desired starting temperature. If the bath is cooled using dry ice, place a suitable amount of powdered dry ice in the insulated tank and slowly add the heat-transfer medium until the tank is filled to a level 30 to 50 mm from the top. If the apparatus is equipped with a liquid nitrogen or CO_2 cooling system and automatic temperature control, follow instructions provided by the manufacturer of the instrument for preparing and operating the bath.~~

~~9.3 Mount the test specimens firmly in the clamping device. Secure the specimens with a torque wrench. To avoid excessive deformation of the specimens, use a torque suitable for the material being tested.~~

~~NOTE 6—It is recommended that a clamping torque of 0.56 ± 0.01 N · m (5 ± 0.1 lb · in.) be used to mount the samples. If slippage of the specimens in the clamp occurs, increase the torque the minimum amount necessary to eliminate the slippage.~~

~~9.4 Mount the clamping device in the testing apparatus and lower the clamping device into the heat-transfer medium. If dry ice is being used as a coolant, maintain constant temperature by the judicious addition of small quantities of dry ice. If the apparatus is equipped with a liquid nitrogen or CO_2 cooling system and automatic temperature control, follow the manufacturer's instructions for setting and maintaining temperature.~~

~~9.5 After waiting for 3 ± 0.5 min, record the temperature and deliver a single impact to the specimens.~~

9.2 ~~Remove the clamping device from the testing apparatus and remove the individual specimens from the clamping device. Allow the specimens to warm up prior to being bent for inspection of cracks by leaving the specimens at room temperature for 1 min or by placing them in lukewarm water for 10 to 15 s. Examine each specimen to determine whether or not it has failed. Failure is defined as the division of a specimen into two or more completely separated pieces or as any crack in the specimen which is visible to the unaided eye. Where a specimen has not completely separated, it shall be bent to an angle of 90° in the same direction as the bend caused by the impact and examined for cracks at the bend. Record the number of failures and the temperature at which they were tested.~~ *Determination of Brittleness Temperature:*

9.2.1 In establishing the brittleness temperature of a material, it is recommended that the test be started at a temperature at which 50 % failure is expected. Test a minimum of ten specimens at this temperature. If all of the specimens fail, increase the temperature of the bath by 10°C and repeat the test using new specimens. If none of the specimens fail, decrease the bath temperature by 10°C and repeat the test using new specimens. If the approximate brittleness temperature is not known, select the start temperature arbitrarily.

9.2.2 Prior to beginning a test, prepare the bath and bring the apparatus to the desired starting temperature. If the bath is cooled using dry ice, place a suitable amount of powdered dry ice in the insulated tank and slowly add the heat-transfer medium until the tank is filled to a level 30 to 50 mm from the top. If the apparatus is equipped with a liquid nitrogen or CO_2 cooling system and automatic temperature control, follow instructions provided by the manufacturer of the instrument for preparing and operating the bath.

9.2.3 Mount the test specimens firmly in the specimen clamp. Secure the specimens with a torque wrench. To avoid excessive deformation of the specimens, use a torque suitable for the material being tested.

9.2.4 Mount the specimen clamp in the testing apparatus and lower the specimen clamp into the heat-transfer medium. If dry ice is being used as a coolant, maintain constant temperature by the judicious addition of small quantities of dry ice. If the apparatus is equipped with a liquid nitrogen or CO_2 cooling system and automatic temperature control, follow the manufacturer's instructions for setting and maintaining temperature.