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Rubber, vulcanized or thermoplastic — Determination of low-temperature brittleness

Caoutchouc vulcanisé ou thermoplastique — Détermination de la fragilité à basse température

Reference number
ISO 812:2006(E)

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 812 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 812:1991), which has been technically revised.

Rubber, vulcanized or thermoplastic — Determination of low-temperature brittleness

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies a method for determining the lowest temperature at which rubber materials do not exhibit brittle failure or the temperature at which half of the test pieces used in a test fail when impacted under specified conditions.

The temperatures thus determined do not necessarily relate to the lowest temperature at which the material can be used since the brittleness will be affected by the conditions of test and especially by the rate of impact. Data obtained by this method should, therefore, be used to predict the behaviour of rubbers at low temperatures only in applications in which the conditions of deformation are similar to those specified in the test.

Three procedures are described:

— procedure A, in which the brittleness temperature is determined;

— procedure B, in which the brittleness temperature for 50 % failure is determined;

— procedure C, in which the test piece is impacted at a specified temperature.

Procedure C is used in the classification of rubber materials and for specification purposes.

NOTE A similar test for rubber-coated fabrics is described in ISO 4646, *Rubber- or plastics-coated fabrics — Low-temperature impact test*.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
brittleness temperature
lowest temperature at which none of a set of of test pieces fractures due to low-temperature embrittlement when tested under the specified conditions

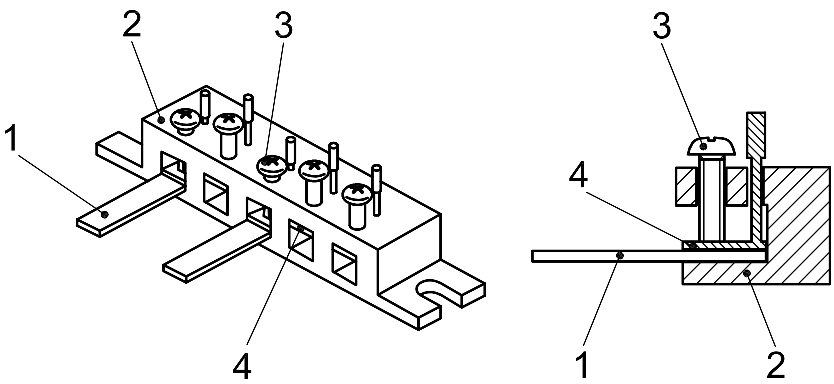
3.2
50 % brittleness temperature
temperature at which 50 % of a set of test pieces fracture due to low-temperature embrittlement when tested under the specified conditions

3.3
testing speed
relative linear velocity at impact between the striking edge of the test apparatus and a clamped test piece.

4 Apparatus and materials

4.1 Test piece clamp and striker, meeting the requirements of 4.1.1 to 4.1.3.

4.1.1 The test piece clamp shall be rigid and designed to hold the test piece(s) as cantilever beam(s). Each individual test piece shall be held firmly and securely in the clamp without distortion. A suitable example of a clamp is shown in Figure 1.



Key

1 test piece

2 body of clamp

3 holding screw

4 test piece holder

Figure 1 — Example of test piece clamp

4.1.2 The striking edge shall move relative to the test piece(s) along a path normal to the upper surface of the test piece(s) at a linear testing speed of 2,0 m/s ± 0,2 m/s at impact. The speed shall be maintained within this range for at least 6 mm of travel following the impact.

In order to obtain a speed within the specified limits during and after impact, care shall be taken to ensure that the striking energy is sufficient. It has been found that a striking energy of at least 3,0 J per test piece is necessary. It may therefore be necessary to limit the number of test pieces impacted at one time.

4.1.3 The principal dimensions of the apparatus [see Figures 2a) and 2b)] shall be as follows:

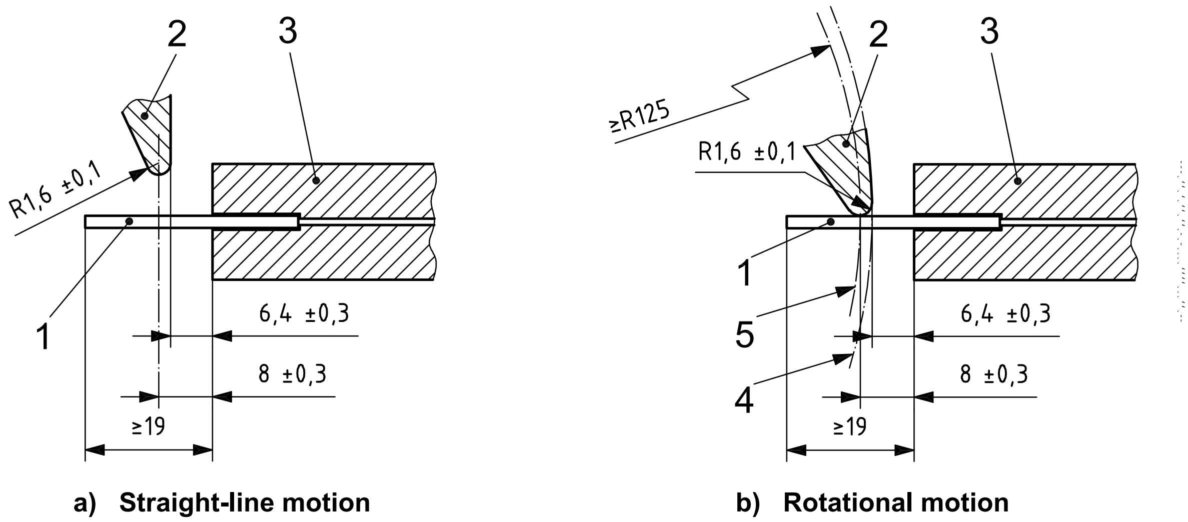
a) the striking edge shall have a radius of 1,6 mm ± 0,1 mm;

b) the clearance between the striker and the test piece clamp at impact shall be 6,4 mm ± 0,3 mm;

c) the separation between the point of impact of the striking edge and the test piece clamp shall be 8 mm ± 0,3 mm.

NOTE Commercial apparatus is available meeting the requirements of this International Standard in which the striking edge is rotated by a motor, or travels in a straight line under the action of a solenoid, gravity or a spring. A method for the speed calibration of a solenoid-actuated-type low-temperature impact tester is given in Annex A.

Dimensions in millimetres



Key

1 test piece
2 striker
3 test piece clamp
4 locus described by point on striker closest to test piece clamp
5 locus described by impact point on striker

Figure 2 — Test piece clamp and striker

4.2 Temperature indicator, comprising a thermocouple, thermometer or other temperature-sensing device capable of covering the range of temperatures in which testing is being carried out and accurate to within ± 0,5 °C.

The temperature sensor shall be placed as near to the test pieces as possible.

4.3 Bath or test chamber, capable of holding the heat-transfer medium (4.4) and of maintaining it at the test temperature to within ± 0,5 °C.

4.4 Heat-transfer medium, liquid or gaseous, which remains fluid at the test temperature and which does not appreciably affect the material being tested.

NOTE The following fluids have been used satisfactorily:

a) for temperatures down to – 60°C, silicone fluids of kinematic viscosity 5 mm^2/s at ambient temperature, which are usually suitable owing to their chemical inertness towards rubbers, their non-flammability and their non-toxicity;

b) for temperatures down to – 70 °C, ethanol;

c) for temperatures down to – 120 °C, methylcyclohexane cooled by liquid nitrogen (found to be satisfactory with the use of suitable apparatus).

5 Test pieces

Test pieces shall be

— either type A: a strip 26 mm to 40 mm long, 6 mm ± 1 mm wide and 2,0 mm ± 0,2 mm thick;

— or type B: a test piece 2,0 mm ± 0,2 mm thick and of the shape and dimensions given in Figure 3.

Test pieces shall be prepared in accordance with ISO 23529. They shall normally be punched from sheet using a suitable sharp die. Alternatively, type A test pieces may be prepared using sharp, parallel double-bladed cutters, in a single stroke. The strip so formed is then cut to the correct length.

Dimensions in millimetres

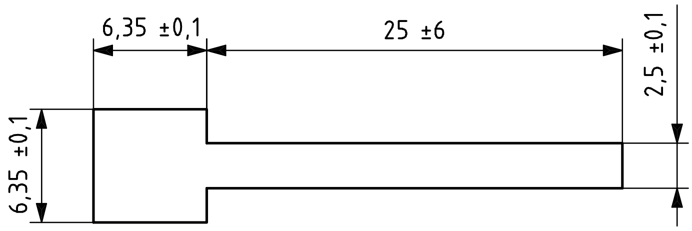


Figure 3 — Type B test piece

6 Time interval between manufacture and testing

Unless otherwise specified, the time interval between the date the material was formed and testing shall be in accordance with ISO 23529.

7 Procedure

7.1 Procedure A (determination of brittleness temperature)

7.1.1 Bring the bath or test chamber to a temperature below the expected lowest temperature of non-failure. In the case of a liquid heat-transfer medium, place sufficient liquid in the bath to ensure approximately 25 mm depth or more of the liquid over the test piece(s). Pre-cool the test piece clamp by immersing it in the cooled bath or test chamber.

7.1.2 Quickly mount the test piece(s) in the test piece clamp and immerse them for 5 min at the test temperature when using a liquid medium or for 10 min when using a gaseous medium (see also ISO 23529).

NOTE 1 For very soft materials, it may be necessary to use a device to support the test piece horizontally until just before the striker is released.

The free length of the test piece(s) shall be greater than 19 mm.

Test five type A or type B test pieces. If the available striking energy has the minimum value specified in 4.1.2, they may all be tested at the same time.

Proper tightening of the clamp is of the utmost importance. The clamp shall be tightened so that each test piece is held with approximately the same clamping torque.

NOTE 2 It has been reported that the temperature at which a test piece fails may be affected by the clamping torque. Clamping to a torque of 0,15 N to 0,25 N is suggested.

7.1.3 After immersion for the specified time at the test temperature, record the temperature and deliver a single impact blow to the test piece(s).

7.1.4 Remove the test pieces from the test piece clamp and allow them to reach standard laboratory temperature. Examine each test piece 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. Where a test piece 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.

7.1.5 Repeat the test at each of a series of successively higher temperatures 10 °C apart, using a new set of test pieces at each temperature, until no failure is obtained. Then decrease the temperature to the highest value at which a failure was observed and carry out tests at temperatures increasing at 2 °C intervals to determine the temperature at which no failure is observed. Record this as the brittleness temperature.

NOTE If crystallization or time-dependent effects of plasticizers are to be studied, longer conditioning periods in a gaseous medium may be used.

7.2 Procedure B (determination of 50 % brittleness temperature)

7.2.1 Carry out the procedure described in 7.1.1 to 7.1.4, except that the starting temperature is that at which 50 % failure is expected.

7.2.2 If all of the test pieces fail at the starting temperature, increase the temperature by 10 °C and repeat the test. If none of the test pieces fail at the starting temperature, decrease the temperature by 10 °C and repeat the test. Increase or decrease the temperature in increments of 2 °C and repeat the test until the lowest temperature at which none of the test pieces fails and the highest temperature at which all of the test pieces fail is determined. Record the number of failures at each temperature. Use a new set of test pieces at each temperature. Determine the 50 % brittleness temperature by calculation using the equation in 7.2.3 or by the graphical method described in 7.2.4.

7.2.3 Calculation: From the number of failures at each temperature, calculate the percentage of failures at each temperature to determine the 50 % brittleness temperature from the following equation:

$$T_{\mathrm{b}} = T_{\mathrm{h}} + \Delta T\left(\frac{S}{100} - \frac{1}{2}\right)$$

where

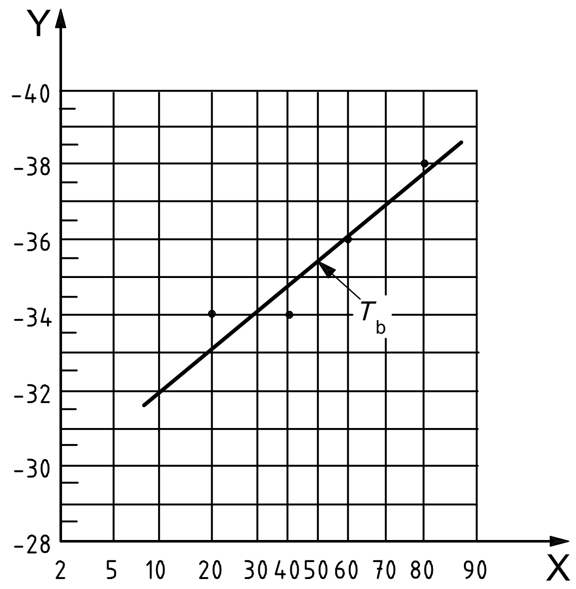
T_{b} is the 50 % brittleness temperature (°C);

T_{h} is the highest temperature at which all test pieces fail (°C);

ΔT is the interval between the test temperatures (°C);

S is the total of the percentages of failures at each temperature, from the temperature at which no test piece fails to the temperature at which they all fail, T_{h} (%).

7.2.4 Graphical method: From the number of failures at each temperature, calculate the percentage of failures at each temperature. Next, using normal probability paper as shown in Figure 4, plot these percentages against the temperature, taking the temperature on the linear scale and the percentage of failures on the probability scale, and draw the best-fit straight line through the points. The temperature at the point of intersection of this line and the 50 % probability line is T_{b}, the 50 % brittleness temperature.



Key

X percentage of failures (%)

Y temperature (°C)

Figure 4 — Determination of 50 % brittleness temperature, T_b, by the graphical method

7.3 Procedure C (testing at a specified temperature)

7.3.1 Carry out the procedure described in 7.1.1 to 7.1.4, except that the temperature used is that specified in the material specification or material classification.

7.3.2 Report the material as satisfactory if no failure is observed in any one of the test pieces, or unsatisfactory if any test pieces have failed.

8 Precision

8.1 General

Precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272:2005, *Rubber and rubber products — Determination of precision for test method standards*. Outliers in the original data were treated at the 5 % and 2 % significance level in accordance with the procedures described in ISO/TR 9272.

8.2 Interlaboratory test programme

8.2.1 An interlaboratory test programme (ITP) was organized by Japan in 2004. Three different compounds of SBR, CR and NBR were used for the brittleness testing. These compounds have different brittleness temperatures.

A total of seven laboratories participated in the ITP, four from Japan and one each from Germany, Thailand and the United Kingdom.

Fully prepared rubber test pieces were sent to each laboratory for evaluation in the ITP, i.e. a type 1 precision was determined.

8.2.2 The brittleness temperature and the 50 % brittleness temperature were determined by the procedures described in Clause 7.

8.2.3 Two types of test piece, type A (strip type) and type B (T-shaped type), were used.

8.3 Precision results

The results of the ITP are given in Table 1. The symbols used in the table are defined as follows:

r is the repeatability, in measurement units;

(r) is the repeatability, in percent (relative);

R is the reproducibility, in measurement units;

(R) is the reproducibility, in percent (relative).

Table 1 — Precision data

Test piece	Test measurement	Material	Mean value (number of results = 7 × 2 = 14)	Within-laboratory (repeatability)		Between laboratories (reproducibility)	
				r	(r)	R	(R)
Type A	Brittleness temperature (°C)	SBR	– 50,60	0,00	0,00	5,52	– 10,90
		CR	– 32,34	2,17	– 6,71	7,78	– 24,07
		NBR	– 26,00	5,17	– 19,87	7,42	– 28,52
	50 % brittleness temperature (°C)	SBR	– 53,64	1,64	– 3,06	5,11	– 9,52
		CR	– 33,93	1,32	– 3,89	5,39	– 15,89
		NBR	– 28,64	2,12	– 7,39	10,31	– 35,99
Type B	Brittleness temperature (°C)	SBR	– 51,30	2,83	– 5,52	12,96	– 25,26
		CR	– 33,32	2,74	– 8,22	7,98	– 23,95
		NBR	– 26,80	4,38	– 16,36	9,34	– 34,86
	50 % brittleness temperature (°C)	SBR	– 55,02	2,70	– 4,91	13,73	– 24,96
		CR	– 35,63	1,83	– 5,15	1,90	– 5,34
		NBR	– 30,15	3,53	– 11,69	11,58	– 38,40

The repeatability of the brittleness temperature of SBR is given as 0 because all the within-laboratory results had the same value after the elimination of outliers.

In terms of the mean values, the 50 % brittleness temperature was, as would be expected, lower than the brittleness temperature. The type B test pieces gave slightly lower temperatures than the type A test pieces.

In terms of the precision obtained, the values obtained for the 50 % brittleness temperature and the brittleness temperature for both type A and type B test pieces were, on average, very similar.

9 Test report

The test report shall include the following information:

a) Sample details:

1) all details necessary for the complete identification of the material tested;

2) the type of test piece used.

b) Test method:

1) the heat-transfer medium and type of test equipment used;

2) whether procedure A, B or C was used.

c) Test details:

1) a reference to this International Standard;

2) the laboratory temperature;

3) the time and temperature of conditioning prior to test;

4) the temperature(s) of test.

d) Test results:

1) the number of test pieces used, and the number impacted at each impact;

2) in the case of procedure A or B, the brittleness temperature or 50 % brittleness temperature, respectively;

3) in the case of procedure C, whether or not the material was satisfactory;

4) the date of the test.

Annex A
(informative)

Speed calibration of a solenoid-actuated low-temperature impact tester[1)]

A.1 Speed calibration prior to testing

A.1.1 Principle

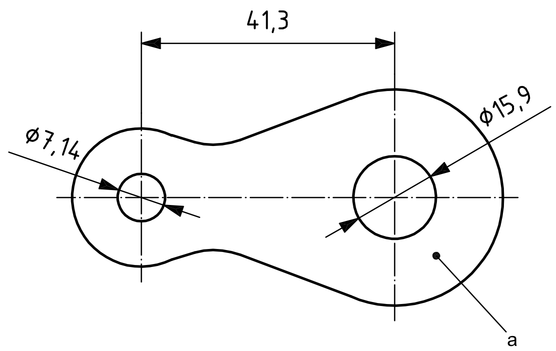
The height, h, to which a steel ball, suspended on the striker mechanism of the tester, rises after the striker has had its upward motion halted by contact with a mechanical stop is measured. The ball decelerates in such a manner that the law governing a body moving freely under the influence of gravity applies.

A.1.2 Procedure

A.1.2.1 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 in the ball support (see Figure A.1) over the guide rod and replace and secure the nut.

Dimensions in millimetres



Key

a Thickness = 3,2 mm.

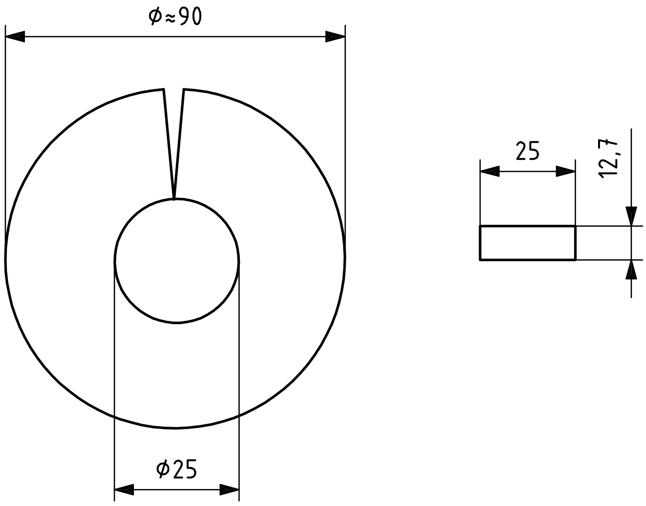
Figure A.1 — Ball support

A.1.2.2 Adjusting stroke or striker

Remove the metal guard from around the solenoid. Spread open the rubber bumper (see Figure A.2) and insert it around the armature. Replace the solenoid guard. Insert a typical test piece 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 is in contact with the test piece but that the bar is not in the plane of the test piece. If the striker bar is not in contact with the test piece, the rubber bumper shall be removed and replaced by a thinner bumper. Conversely, if the striker bar moves into the plane of the test piece, the bumper shall be replaced by a thicker one.

1) This annex applies only to certain types of impact-testing apparatus.

Dimensions in millimetres



Hardness of bumper ≈ 70 IRHD

Figure A.2 — Rubber bumper

A.1.2.3 Placement of ball and measuring tube

Place a 19 mm 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 in a vertical position directly over the ball. The tube should contain a scale divided into 5 mm 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.

A.1.2.4 Measurement and calculation

With the tester equipped as described above and devoid of test pieces and immersion medium, actuate the solenoid and read the ball height to the nearest 5 mm. Make at least five measurements. Average all results and convert the average to metres. Determine the striker speed, v, in metres per second, from the following equation:

$$v = \sqrt{2gh}$$

where

g is the acceleration due to gravity, in metres per second squared (= 9,8 m/s^2);

h is the average ball height, in metres.

NOTE 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.

A.2 Speed calibration during testing

A.2.1 With the tester equipped with ball support, ball and measuring tube (see A.1), but without the rubber bumper (tested in normal operating condition) and devoid of test pieces and immersion medium, actuate the solenoid and read the ball height to the nearest 5 mm. Make ten measurements. From the lowest and highest ball height readings, determine the range in striker speed, using the equation in A.1.2.4. This range is termed “range of speed at the top of the stroke”.

A.2.2 With the tester equipped as described in A.2.1, but also with test piece(s) and immersion medium, conduct the brittleness test as described in Clause 7. Read the ball height each time the solenoid is actuated. Convert the ball height to speed as shown in A.1.2.4. If the speed lies within the predetermined range of speeds at the top of the stroke, the test should be considered valid. If the speed lies outside the predetermined range, the test is invalid and the result 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 test pieces tested per impact.

A.2.3 The following example typifies the entire speed calibration procedure for solenoid-actuated testers.

a) Using the procedure specified in A.1, the striker speed at the point of impact of a tester devoid of test pieces and immersion medium was found to be 1,9 m/s. This speed is within the limits specified in 4.1.2.

b) Using the procedure in A.2.1, with the tester devoid of test pieces and immersion medium, the range of striker speeds at the top of the stroke was found to be 2,5 m/s to 2,7 m/s. This range becomes the acceptable range for this series of tests. The acceptable range should be established each time the striker speed at the point of impact is determined (see A.1).

c) Using the procedure of A.2.2, with the tester containing test piece(s) and immersion medium, the speed at the top of the stroke during the first solenoid actuation was found to be 2,5 m/s. The speed is within the acceptable range and the test is valid.

d) The speeds at the top of the stroke during the second and third solenoid actuations were found to be 2,4 m/s and 2,3 m/s, respectively. These speeds are outside the acceptable range and both tests are invalid.

e) Adjustments were made to increase the speed at the top of the stroke, using the procedure given in A.2.2.

f) The speeds at the top of the stroke during the fourth and all subsequent solenoid actuations were found to lie between 2,5 m/s and 2,7 m/s. The results of all these tests are valid.

ICS 83.060

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