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Flexible cellular polymeric materials — Determination of hardness (indentation technique)

Matériaux polymères alvéolaires souples — Détermination de la dureté (technique par indentation)

Reference number
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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 2439 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 4, *Products (other than hoses)*.

This fourth edition cancels and replaces the third edition (ISO 2439:1997 and ISO 2439:1997/Cor.1:1998), which has been technically revised.

Major modifications in this revised text are:

a) change in Scope to cover five methods;

b) inclusion of Figure 1 to illustrate the force-indentation curve; and

c) inclusion of informative annexes.

Flexible cellular polymeric materials — Determination of hardness (indentation technique)

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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

The indentation hardness of flexible cellular materials is a measure of their load-bearing properties. This International Standard specifies four methods (A to D) for the determination of indentation hardness and one method (E) for determination of compressive deflection coefficient and hysteresis loss rate of flexible cellular materials. Annex A provides a summary of test parameters and typical force-indentation graphs obtained with these methods.

These five methods are applicable only to latex foam, urethane foam and PVC foam of the open-cell type. The methods specified can be used for testing finished articles and for the characterization of bulk material.

This International Standard specifies the following methods:

a) Method A — Determination of the 40 %/30 s indentation hardness index, which gives a single indentation measurement for laboratory test purposes;

b) Method B — Determination of the 25 %-40 %-65 %/30 s indentation hardness characteristics, which provides information about the shape of the hardness indentation curve;

c) Method C — Determination of the 40 % indentation hardness check, which is a quick procedure suitable for quality control testing;

d) Method D — Determination of the 25 %/20 s low indentation hardness index, which is a quick procedure suitable as an inspection test;

e) Method E — Determination of the compressive deflection coefficient and hysteresis loss rate, which gives additional information about the load-bearing properties of materials.

The results obtained by these methods relate only to the test conditions specified and cannot, in general, be used directly for design purposes.

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 1382, *Rubber — Vocabulary*

ISO 7500-1, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

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 terms and definitions given in ISO 1382 and the following apply.

3.1
indentation hardness
total force required to produce, under specified conditions, a specified indentation of a standard test piece

NOTE Indentation hardness is expressed in newtons.

3.2
compressive deflection coefficient
S_f
ratio of the 65 % indentation force deflection to the 25 % indentation force deflection

3.3
hysteresis loss rate
A_f
energy difference between the loading and unloading of a test piece under cyclic deformation

NOTE Hysteresis loss rate is expressed as a percentage of the loading energy.

4 Principle

The forces required to produce specified indentations under specified conditions are measured.

5 Apparatus

5.1 Test machine.

The test machine shall be capable of indenting the test piece between a supporting surface (5.2) and an indentor (5.3) having a uniform relative motion, in the vertical direction, of (100 ± 20) mm/min.

The test machine shall have means of measuring force in conformance with Class 1 of ISO 7500-1 or of measuring with a precision of ± 1 N, and of measuring the test piece thickness under load with a precision of ± 0,25 mm.

The test machine for Method C and Method E shall have its force gauge fitted with a tell-tale needle and/or shall be equipped to make autographic load-indentation plots.

The test machine shall also be capable of maintaining the specified degree of indentation with a precision of ± 0,25 mm for the specified period.

5.2 Supporting surface.

Unless otherwise specified, the test pieces shall be supported on a smooth, flat, horizontal and rigid surface larger than the test piece and suitably vented with holes approximately 6 mm in diameter and of approximately 20 mm pitch, to allow the escape of air from below the test piece.

5.3 Indentor.

The indentor shall be mounted preferably by a ball joint free from vertical movement, although other methods of mounting are permitted. The indentor shall be flat and circular, with a diameter of 200^{+3}_{0} mm and a $1{,}0^{+0{,}5}_{0}$ mm radius at the lower edge. The lower surface shall be smooth but not polished.

6 Test pieces

6.1 Shape and dimensions

Material shall be cut to obtain a standard-size square of length of side 380^{+20}_{0} mm, with a thickness of (50 ± 2) mm. Sheets of less than this standard thickness shall be plied together to approximate as closely as possible to the standard thickness.

Finished articles may be tested as agreed between purchaser and supplier.

NOTE Results on plied material and on finished articles may not be the same as would be obtained with the standard test piece.

6.2 Samples showing orientation

If samples show orientation of the cellular structure, the direction in which the indentation is to be carried out shall be agreed between the interested parties. Normally, testing should be carried out in that direction in which the finished product will be stressed under service conditions.

6.3 Conditioning

Material shall not be tested sooner than 72 h after manufacture, unless at either 16 h or 48 h after manufacture it can be demonstrated that the mean result does not differ by more than ± 10 % from that obtained after 72 h. Testing is permitted at either 16 h or 48 h if, at the specified time, the above criterion has been satisfied.

Prior to the test, the test pieces shall be conditioned, undeflected and undistorted, for at least 16 h in one of the following atmospheres, as given in ISO 23529.

— (23 ± 2) °C, (50 ± 5) % relative humidity;

— (27 ± 2) °C, (65 ± 5) % relative humidity.

This conditioning period can form the latter part of the period following manufacture.

In case of quality control tests, test pieces may be sampled at 12 h after manufacture or later, and testing is permitted after conditioning for at least 6 h in one of the specified atmospheres.

7 Procedure

7.1 General

Carry out the test immediately after conditioning, preferably under the same atmospheric conditions as specified in 6.3.

NOTE See Annex A for assistance in understanding each test method.

Position the test piece on the supporting surface so that the centre of the test piece, or other agreed test area, is located below the centre of the indentor. Test pieces having cavities on one side shall be placed with the cavity side next to the supporting surface.

If a test piece has cavities, the acceptable characteristics of the cavities, such as quantity, dimensions and location in the test piece, should be agreed between purchaser and supplier.

7.2 Preliminary indentation for Methods A, B and C

a) Apply a force of $5_{-2}^{\ 0}$ N to the selected test area and measure the thickness of the test piece. This value is the point of zero indentation.

b) Indent the test piece at an indentor rate of (100 ± 20) mm/min, to produce an indentation of (70 ± 2,5) % of the thickness. After reaching this deflection, release the load at the same rate.

c) Repeat this loading and unloading twice more, then proceed with 7.3, 7.4 or 7.5 as appropriate.

7.3 Method A — Determination of the 40 %/30 s indentation hardness index

Immediately after the third unloading [see 7.2 c)], indent the test piece by (40 ± 1) % of its thickness. Maintain this deflection for a period of (30 ± 1) s, note the corresponding force, in newtons, and release the force.

Only the result of a test conducted by Method A, on a standard-size test piece without plying, shall be known as the indentation hardness index.

7.4 Method B — Determination of the 25 %-40 %-65 %/30 s indentation hardness characteristics

Immediately after the third unloading [see 7.2 c)], carry out the following operations:

a) indent the test piece by (25 ± 1) % of the thickness;

b) maintain this indentation for a period of (30 ± 1) s;

c) measure the force required;

d) increase the indentation to (40 ± 1) % of the thickness;

e) maintain this indentation for a period of (30 ± 1) s;

f) measure the force required;

g) increase the indentation to (65 ± 1) % of the thickness;

h) maintain this indentation for a period of (30 ± 1) s;

i) measure the force required.

The results of a test conducted by Method B on a standard test piece shall be known as the standard indentation hardness characteristics of that material. If a product is tested, the results shall be known as the product indentation hardness characteristics.

NOTE Convenient means of expressing the results obtained by Method B are indentation factors, which are the ratios of the forces required to obtain the indentations of 25 % and 65 % to the force required to obtain the indentation of 40 %.

7.5 Method C — Determination of the 40 % indentation hardness check

Immediately after the third unloading [see 7.2 c)], carry out the following operations:

a) start the autographic recording, or bring back the tell-tale needle of the force gauge, and indent the test piece to (40 ± 1) % of its thickness;

b) record the force, in newtons, using the tell-tale needle or the instantaneous maximum of the autographic recorder;

c) release the force.

The results of a test conducted by Method C shall be known as the indentation hardness check.

NOTE This is a faster, quality-control test for indentation hardness. The variability of results obtained in this way is usually higher. It should also be noted that the results obtained in this way may be related to results obtained with Method A but are usually higher.

7.6 Method D — Determination of the 25 %/20 s low indentation hardness index

7.6.1 Preliminary indentation

a) Apply a force of $5^{\ 0}_{-2}$ N to the selected test area and measure the thickness of the test piece. This value is the point of zero indentation.

b) Indent the test piece at an indentor rate of (100 ± 20) mm/min to produce an indentation of (75 ± 2,5) % of its thickness. After reaching this deflection, release the load at the same rate.

7.6.2 Measuring

Immediately after the unloading [see 7.6.1 b)], indent the test piece by (25 ± 1) % of its thickness. Maintain this deflection for a period of (20 ± 1) s, note the corresponding force, in newtons, and release the force.

Only the result of a test conducted by Method D, on the standard-size test piece without plying, shall be known as the low indentation hardness index.

7.7 Method E — Determination of the compressive deflection coefficient and hysteresis loss rate

7.7.1 Preliminary indentation

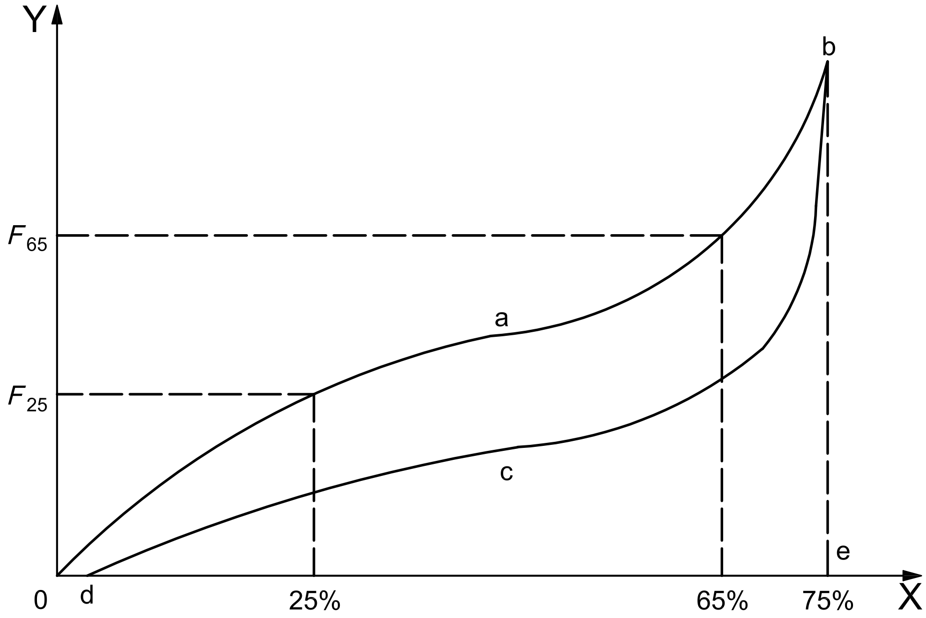
a) Apply a force of $5^{\ 0}_{-2}$ N to the selected test area and measure the thickness of the test piece. This value is the point of zero indentation.

b) Indent the test piece at an indentor rate of (100 ± 20) mm/min to produce an indentation of (75 ± 2,5) % of its thickness. After reaching this deflection, release the load at the same rate.

c) Allow the test piece to rest for (4 ± 1) min.

7.7.2 Measuring

Immediately after the rest period [see 7.7.1 c)], indent the test piece at an indentor rate of (100 ± 20) mm/min, to produce an indentation of (75 ± 2,5) % of the thickness as measured in 7.7.1 a), and simultaneously record the force-indentation curve. After reaching (75 ± 2,5) % indentation, release the force at the same rate and complete a whole force-indentation curve as illustrated in Figure 1. The time interval between completion of the compression cycle and commencement of the decompression cycle shall not exceed 2 s.



Key

X indentation, %

Y force, F

a typical line for compression cycle

b top point

c typical line for decompression cycle

d end point

e point of 75 % indentation of the test piece

Figure 1 — Typical force-indentation curve

7.7.3 Expression of results

7.7.3.1 Compressive deflection coefficient

The compressive deflection coefficient, S_f, is given by the equation:

$$S_f = \frac{F_{65}}{F_{25}}$$

where

F_{25} is the force at 25 % indentation in compression, in newtons;

F_{65} is the force at 65 % indentation in compression, in newtons.

7.7.3.2 Hysteresis loss rate

The hysteresis loss rate, A_f (%), is given by the equation:

$$A_f = \frac{\text{Area 0abcd0}}{\text{Area 0abe0}} \times 100$$

where

Area 0abcd0 is the area contained within the hysteresis curve 0abcd0 (see Figure 1);

Area 0abe0 is the area under the curve 0ab (see Figure 1).

8 Repeat tests

For repeat tests on the same test piece, a minimum recovery period of 16 h shall be observed.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) the method used and the type of results obtained (e.g. product indentation hardness characteristics);

c) the temperatures and relative humidities of conditioning and testing;

d) whether bulk material or finished articles were tested;

e) the dimensions of the test piece and, in particular, the thickness as determined in 7.2 a);

f) where applicable, the number of plies constituting the test piece;

g) whether skins were present and, if so, how many;

h) the indentation hardness(es): values up to 100 N shall be quoted to the nearest unit; values over 100 N shall be quoted to the nearest 5 N;

i) any deviations from this International Standard.

Annex A
(informative)

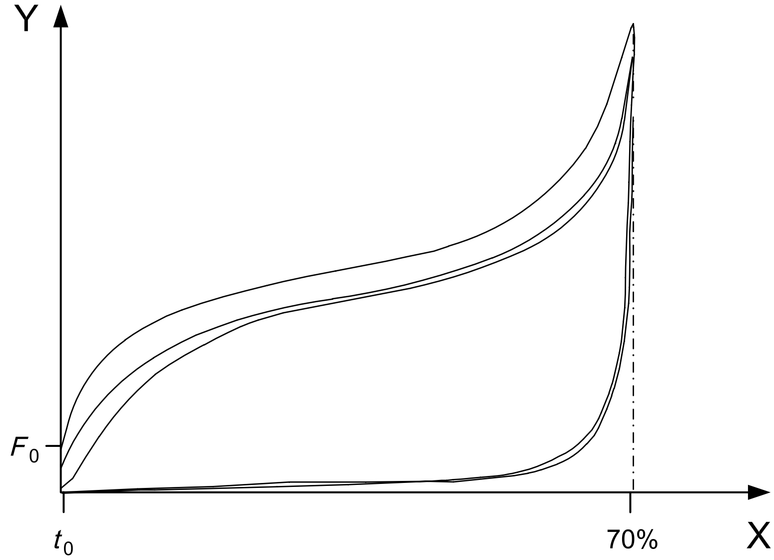
Test method parameters and typical graphs

A.1 Test parameters for Methods A, B, C, D and E

Table A.1 — Parameters of test methods

<table>
<tr><th>Test parameters</th><th>Method A</th><th colspan="3">Method B</th><th>Method C</th><th>Method D</th><th>Method E [a]</th></tr>
<tr><td>Number of preliminary indentations</td><td>3</td><td colspan="3">3</td><td>3</td><td>1</td><td>1</td></tr>
<tr><td>Preliminary indentation, % of test piece thickness</td><td>70 ± 2,5</td><td colspan="3">70 ± 2,5</td><td>70 ± 2,5</td><td>75 ± 2,5</td><td>75 ± 2,5</td></tr>
<tr><td>Rest time after preliminary indentation, min</td><td>—</td><td colspan="3">—</td><td>—</td><td>—</td><td>4 ± 1</td></tr>
<tr><td rowspan="3">Indentation, % of test piece thickness at measurement</td><td>—</td><td>25 ± 1</td><td></td><td></td><td>—</td><td>25 ± 1</td><td></td></tr>
<tr><td>40 ± 1</td><td></td><td>40 ± 1</td><td></td><td>40 ± 1</td><td>—</td><td rowspan="2">0~75~0</td></tr>
<tr><td>—</td><td></td><td></td><td>65 ± 1</td><td>—</td><td>—</td></tr>
<tr><td>Compression hold period before measuring, s</td><td>30 ± 1</td><td>30 ± 1</td><td>30 ± 1</td><td>30 ± 1</td><td>0</td><td>20 ± 1</td><td>—</td></tr>
<tr><td>Indentation hardness symbol</td><td>$HA_{(40\%/30s)}$</td><td>$HB_{(25\%/30s)}$</td><td>$HB_{(40\%/30s)}$</td><td>$HB_{(65\%/30s)}$</td><td>$HC_{(40\%/0s)}$</td><td>$HD_{(25\%/20s)}$</td><td>—</td></tr>
<tr><td colspan="8">[a] See 7.7.3.</td></tr>
</table>

A.2 Sample force-indentation graphs using Methods A, B, C and D



Key

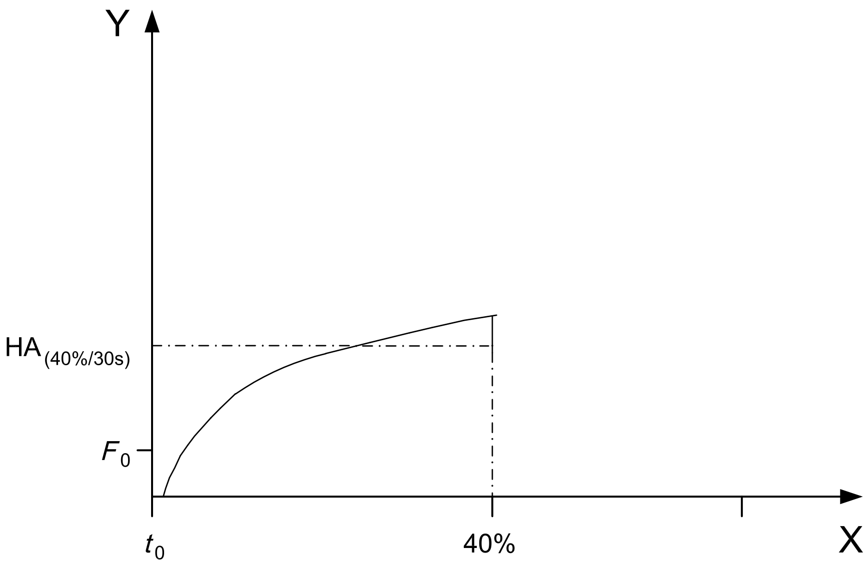
X indentation %

Y force, F

F_0 5_{-2}^{0} N, preload force at which initial thickness is measured

t_0 initial thickness of test piece

Figure A.1 — Sample force-indentation graph of preliminary indentation, Method A, B or C



Key

X indentation, %

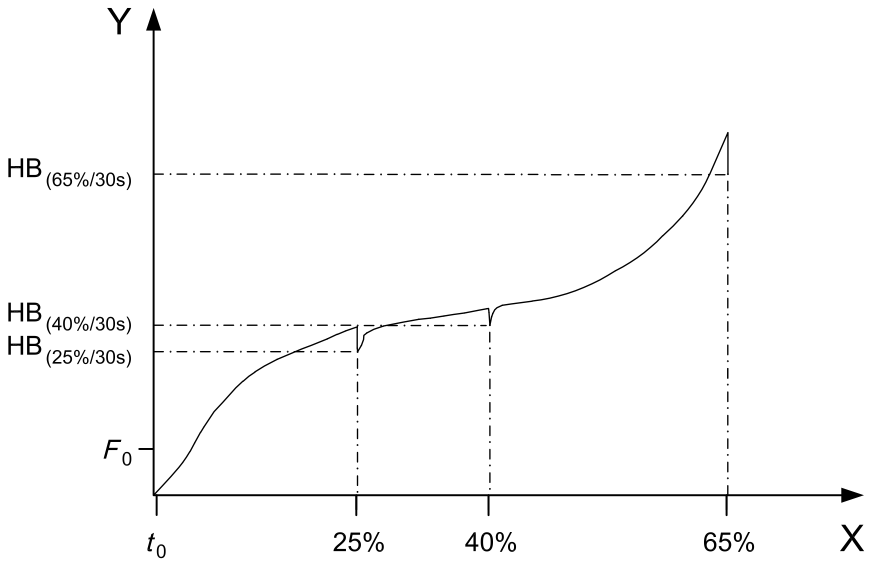
Y force, F

F_0 5_{-2}^{0} N, preload force at which initial thickness is measured

t_0 initial thickness of test piece

HA hardness measured according to Method A

Figure A.2 — Sample force-indentation graph using Method A



Key

X indentation, %

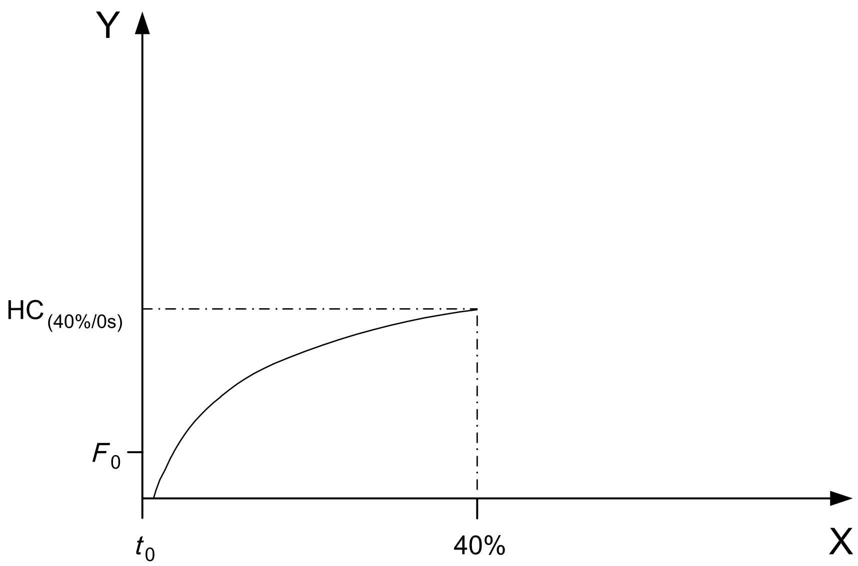
Y force, F

F_0 $5_{-2}^{\ 0}$ N, preload force at which initial thickness is measured

t_0 initial thickness of test piece

HB hardness measured according to Method B

Figure A.3 — Sample force-indentation graph using Method B



Key

X indentation, %

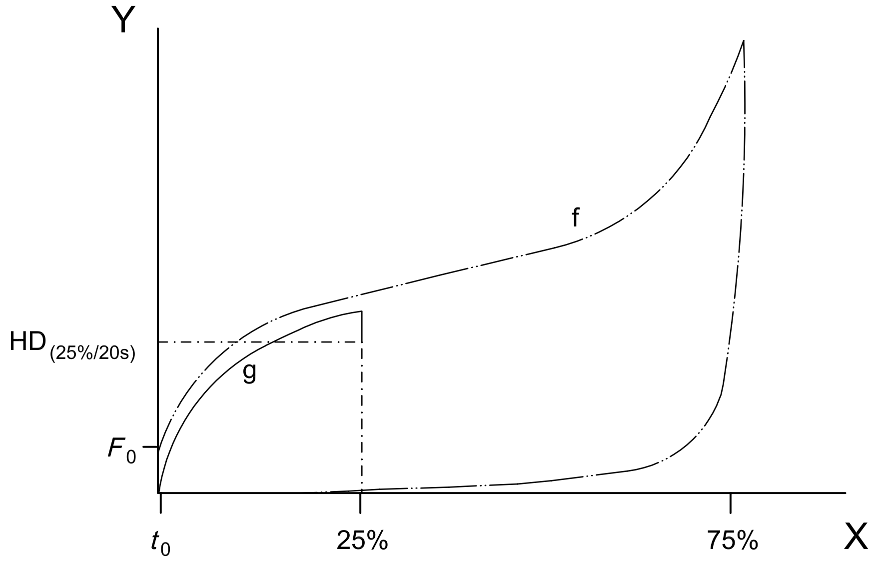
Y force, F

F_0 $5_{-2}^{\ 0}$ N, preload force at which initial thickness is measured

t_0 initial thickness of test piece

HC hardness measured according to Method C

Figure A.4 — Sample force-indentation graph using Method C



Key

X indentation, %

Y force, F

F_0 $5_{-2}^{\ 0}$ N, preload force at which initial thickness is measured

f preliminary indentation curve

g test indentation curve

t_0 initial thickness of test piece

HD hardness measured according to Method D

Figure A.5 — Sample force-indentation graph using Method D

Annex B
(informative)

Precision of Method E

B.1 General

The precision of Method E was determined in accordance with ISO/TR 9272. The precision results as determined by this ITP (inter-laboratory test programme) should not be used for acceptance or rejection of any group of materials without documentation that the results of this precision evaluation are actually applicable to the particular group of materials tested.

B.2 Details

The ITP for precision evaluation was organized by Japan and conducted in 2004. Seven laboratories participated in this ITP using three types of flexible polyurethane foam with different resilience levels.

The compressive deflection coefficient and hysteresis loss rate were measured using Method E in accordance with this International Standard.

B.3 Precision results

B.3.1 General

The precision results for three types of test piece with different resilience levels are given in Table B.1. Three test pieces were tested for each resilience level, and both properties were measured in accordance with the test procedure in Method E.

B.3.2 Repeatability

The repeatability, or local domain precision, for this test method was established from the values in Table B.1 for each measurement parameter. Test results obtained using Method E of this International Standard that differ by more than the tabulated values for r, in units of measurement, and (r), in percent, should be considered as suspect, i.e. to have come from different populations, and suggest that some appropriate investigative action be taken.

B.3.3 Reproducibility

The reproducibility, or global domain precision, for this test method was established from the values in Table B.1 for each measurement parameter. Test results obtained in different laboratories using Method E of this International Standard that differ by more than the tabulated values for R, in units of measurement, and (R), in percent, should be considered as suspect, i.e. to have come from different populations, and suggest that some appropriate investigative action be taken.

Table B.1 — Precision results

Test piece	Property	Mean value	Within laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
Conventional foam	Compressive deflection coefficient	1,78	0,035 2	0,100	5,58	0,069 4	0,196	11,0
	Hysteresis loss rate	44,46	0,959	2,713	6,10	2,324	6,58	14,79
Low resilience foam	Compressive deflection coefficient	2,15	0,044 4	0,126	5,85	0,120	0,337	15,69
	Hysteresis loss rate	67,91	1,787	5,06	7,45	6,314	17,68	26,03
High resilience foam	Compressive deflection coefficient	2,29	0,047	0,132	5,75	0,078	0,221	9,62
	Hysteresis loss rate	33,43	0,349	0,988	2,96	2,844	8,050	24,08

Bibliography

[1] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

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