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Plastics — Determination of puncture impact behaviour of rigid plastics —

Part 2: Instrumented impact testing

Plastiques — Détermination du comportement des plastiques rigides perforés sous l'effet d'un choc —

Partie 2: Essais de choc instrumentés



Reference number ISO 6603-2:2023(E)



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical behavior*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 6603-2:2000), which has been technically revised.

The main changes are as follows:

- references to ISO 6603-1 were replaced by the corresponding text;
- normative references and bibliography were updated and completed;
- requirements for force measurement accuracy were revised;
- definitions for conditioning and test climate were updated;
- testing in a clamped situation were defined as the preferred method;
- precision data was added to <u>Annex G</u>.

A list of all parts in the ISO 6603 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at <u>www.iso.org/members.html</u>.

Plastics — Determination of puncture impact behaviour of rigid plastics —

Part 2: Instrumented impact testing

1 Scope

This document specifies a test method for the determination of puncture impact properties of rigid plastics, in the form of flat specimens, using instruments for measuring force and deflection. It is applicable if a force-deflection or force-time diagram, recorded at nominal constant striker velocity, is necessary for detailed characterization of the impact behaviour.

The test method is applicable to specimens with a thickness between 1 mm to 4 mm.

The method is suitable for use with the following types of material:

- rigid thermoplastic moulding and extrusion materials, including filled, unfilled and reinforced compounds and sheets;
- rigid thermosetting moulding and extrusion materials, including filled and reinforced compounds, sheets and laminates;
- fibre-reinforced thermoset and thermoplastic composites incorporating unidirectional or multidirectional reinforcements such as mats, woven fabrics, woven rovings, chopped strands, combination and hybrid reinforcements, rovings, milled fibres and sheets made from pre-impregnated materials (prepregs).

The method is also applicable to specimens which are either moulded or machined from finished products, laminates and extruded or cast sheet.

The test results are comparable only if the conditions of preparation of the specimens, their dimensions and surfaces as well as the test conditions are the same. In particular, results determined on specimens of different thickness cannot be compared with one another (see <u>Annex E</u>). Comprehensive evaluation of the reaction to impact stress can be obtained by determinations made as a function of impact velocity and temperature for different material variables, such as crystallinity and moisture content.

The impact behaviour of finished products cannot be predicted directly from this test, but specimens may be taken from finished products (see above) for tests by this method.

Test data developed by this method is not intended to be used for design calculations. However, information on the typical behaviour of the material can be obtained by testing at different temperatures and impact velocities (see <u>Annex D</u>) by varying the thickness (see <u>Annex E</u>) and by testing specimens prepared under different conditions.

It is not the purpose of this document to give an interpretation of the mechanism occurring on every particular point of the force-deflection diagram. These interpretations are a task for scientific research.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 6603-2:2023(E)

ISO 291, Plastics — Standard atmospheres for conditioning and testing

ISO 293, Plastics — Compression moulding of test specimens of thermoplastic materials

ISO 294-3, Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates

ISO 295, Plastics — Compression moulding of test specimens of thermosetting materials

ISO 1268-1, Fibre-reinforced plastics — Methods of producing test plates — Part 1: General conditions

ISO 2602, Statistical interpretation of test results — Estimation of the mean — Confidence interval

ISO 2818, Plastics — Preparation of test specimens by machining

ISO 16012, Plastics — Determination of linear dimensions of test specimens

ISO 20753, Plastics — Test specimens

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at <u>https://www.electropedia.org/</u>

3.1

impact velocity

 v_0

velocity of the striker relative to the support at the moment of impact

Note 1 to entry: Impact velocity is expressed in metres per second (m/s).

3.2

force

F

force exerted by the striker on the test specimen in the direction of impact

Note 1 to entry: Force is expressed in newtons (N).

3.3 deflection

1

relative displacement between the striker and the specimen support, starting from the first contact between the striker and the test specimen

Note 1 to entry: Deflection is expressed in millimetres (mm).

3.4 energy

Ε

energy expended in deforming and penetrating the test specimen up to a deflection l

Note 1 to entry: Energy is expressed in joules (J).

Note 2 to entry: Energy is measured as the integral of the force-deflection curve starting from the point of impact up to a deflection l.

3.5 maximum force $F_{\rm M}$ maximum force which occurs during the test

Note 1 to entry: See <u>Figures 1</u> to <u>4</u>.

Note 2 to entry: Maximum force is expressed in newtons (N).

3.6 deflection at maximum force

 $l_{\rm M}$ deflection that occurs at maximum force $F_{\rm M}$

Note 1 to entry: See <u>Figures 1</u> to <u>4</u>.

Note 2 to entry: Deflection at maximum force is expressed in millimetres (mm).

3.7

energy to maximum force

 $E_{\rm M}$ energy expended up to the deflection $l_{\rm M}$ at maximum force

Note 1 to entry: See <u>Figures 1</u> to <u>4</u>.

Note 2 to entry: Energy to maximum force is expressed in joules (J).

3.8 puncture deflection $l_{\rm P}$

deflection at which the force has dropped to half the maximum force $F_{\rm M}$

Note 1 to entry: See Figures 1 to 4 and Note 3 to entry of 3.9.

Note 2 to entry: Puncture deflection is expressed in millimetres (mm).

3.9

puncture energy $E_{\rm P}$ energy expended up to the puncture deflection $l_{\rm P}$

Note 1 to entry: See <u>Figures 1</u> to <u>4</u>.

Note 2 to entry: Puncture energy is expressed in joules (J).

Note 3 to entry: When testing tough materials, a transducer mounted at some distance from the impacting tip may record frictional force acting between the cylindrical part of the striker and the punctured material. The corresponding frictional energy shall not be included in the puncture energy, which, therefore, is restricted to that deflection, at which the force drops to half the maximum force $F_{\rm M}$.

3.10 impact failure

mechanical behaviour of the material under test which may be either one of the following types:

- a) YD yielding followed by deep drawing, see Figure 1
- b) YS yielding followed by an at least partially stable cracking, see Figure 2
- c) YU yielding followed by unstable cracking, see Figure 3
- d) NY no yielding, see Figure 4

Note 1 to entry: The classification of the type of failure shall take into account the shape of the curve as well as the assessment of the broken specimen.

Note 2 to entry: Yielding is characterised by a zero slope at maximum force in the force deflection diagram, or by whitening of the material in the area of puncture, or by a significant reduction of the material thickness in the area where the break appears.

Note 3 to entry: Comparison of Figures 2 and 3 shows puncture deflection l_P and puncture energy E_P are identical for the failure types YS and YU. As shown in Figure 4, identical values at maximum and at puncture are found for the deflection as well as the energy in the case of failure type NY. For complex behaviour see <u>Annex A</u>.

Note 4 to entry: For more guidance on the classification of failure types, see the informative <u>Annex F</u>.



Key

X deflectionY force

Figure 1 — Example of force-deflection diagram for failure by yielding (zero slope at maximum force) followed by deep drawing, and typical appearance of specimens after testing (with lubrication)



- X deflection
- Y force

Figure 2 — Example of force-deflection diagram for failure by yielding (zero slope at maximum force) followed by stable crack growth, and typical appearance of specimens after testing (with lubrication)



X deflection

Y force

NOTE Natural vibration of the force measurement system appears after unstable cracking (striker and load cell).

Figure 3 — Example of force-deflection diagram for failure by yielding (zero slope at maximum force) followed by unstable crack growth, and typical appearance of specimens after testing (with lubrication)



- X deflection
- Y force

Figure 4 — Example of force-deflection diagram for failure without yielding followed by unstable crack growth, and typical appearance of specimens after testing (with lubrication)

4 Principle

The test specimen is punctured at its centre using a lubricated striker, perpendicularly to the testspecimen surface and at a nominally uniform velocity. The resulting force-deflection or force-time diagram is recorded electronically. The test specimen should be clamped in position (preferred) during the test.

The force-deflection diagram obtained in these tests records the impact behaviour of the specimen from which several features of the behaviour of the material can be inferred.

5 Apparatus

5.1 **Testing instrument**, consisting of the following essential components:

- energy carrier, which can be falling mass type or hydraulic type (see <u>5.1.1</u>);
- striker, which shall be lubricated;
- specimen support with a recommended clamping device.

The test device shall permit the test specimen to be punctured at its centre, perpendicular to its surface at a nominally constant velocity. The force exerted on the test specimen in the direction of impact and the deflection from the centre of the test specimen in the direction of impact shall be derivable or measurable (see Figure 5).

The term "falling mass type energy carrier" covers all types of instruments working by the principle of an inertial mass, independent from the direction of movement.

5.1.1 Energy carrier, with a preferred impact velocity v_0 of $(4,4 \pm 0,2)$ m/s (see <u>3.1</u> and note to <u>3.1</u>). To avoid results, which cannot be compared due to the viscoelastic behaviour of the material under impact, the decrease of velocity during the test shall not be greater than 20 %.

NOTE For brittle materials, an impact velocity of 1 m/s can be more appropriate because it reduces the level of vibration and noise and improves the quality of the force-deflection diagram (see <u>Annex A</u>).

5.1.1.1 Hydraulic type, consisting of a high-speed testing machine with suitable attachments.

Any deviation of the velocity of the striker relative to the support during impact shall be controlled, for example by recording deflection-time curves and checking the slope.

5.1.1.2 Inertial-mass type, which may be accelerated. Suitable devices are falling-dart machines.

In the case of a gravitationally accelerated mass and neglecting frictional losses; the impact velocity v_0 corresponds to a drop height H₀ of the energy carrier of (1,0 ± 0,1) m.

For all inertial-mass-type energy carriers the impact velocity shall be measured by velocity-measuring sensors placed close to the point of impact. The maximum decrease of velocity during test results in the minimum mass, $m_{\rm C}$, of the carrier according to Formulae (1) and (2) (see NOTE).

$$m_{\rm c} \ge 6E^* / v_0^2 \tag{1}$$

$$m_{\rm c} \ge 0.31E^* \text{ for } v_0 = 4.4 \text{ m/s}$$
 (2)

where

 $m_{\rm c}$ is the mass of the energy carrier, expressed in kilograms;

- E^* is the highest puncture energy to be measured, expressed in joules (see <u>3.9</u>);
- v_0 is the impact velocity (4,4 m/s, see <u>3.1</u>).

NOTE In many cases, a weighted energy carrier with a total mass m_c of 20 kg has been found to be sufficient for the larger striker and of 5 kg for the smaller striker (see <u>5.1.2</u>).

5.1.2 Striker, preferably having a polished hemispherical striking surface of diameter $(20,0 \pm 0,2)$ mm. Alternatively, a $(10,0 \pm 0,1)$ mm diameter striking surface should be used.

NOTE 1 The size and dimensions of the striker and condition of the surface will affect the impact results.

The striker shall be made of any material with sufficient resistance to wear and of sufficiently high strength to prevent plastic deformation. In practice, hardened steel or materials with lower density (i.e. titanium) have been found acceptable.

The hemispherical surface of the striker shall be lubricated to reduce any friction between the striker and the test specimen (see NOTE 2 and <u>Annex B</u>).

NOTE 2 Test results obtained with a lubricated or dry striker are likely to be different. Below ambient temperatures, condensation can act as a lubricant.

The load cell shall be located within one striker diameter from the tip of the striker, i.e. mounted as closely as possible to the tip to minimize all extraneous forces and sufficiently near to fulfil the frequency-response requirement (see 5.2). An example is shown in Figure 5.

5.1.3 Support ring (see <u>Figures 5</u> and <u>6</u>), placed on a rigid base and designed such that air cannot be trapped under the test specimen, thus avoiding a possible spring effect. Below the support ring, there shall be sufficient space for the striker to travel after total penetration of the test specimen. The

recommended inside diameter of the support ring is (40 ± 2) mm, or alternatively (100 ± 5) mm, with a minimum height of 12 mm.

5.1.4 Base for test device, firmly mounted to a rigid structure so that the mass of the base (see <u>Figure 5</u>) is of sufficient stiffness to minimize deflection of the specimen support.

When calculating the deflection from the kinetics of the accelerated mass, a minimum mass ratio $m_{\rm B}/m_{\rm C}$ of 10 between base ($m_{\rm B}$) and energy carrier ($m_{\rm C}$) shall be used. This prevents the base from being accelerated by more than 1 % of the impact speed up to the end of the test. For directly measured deflections, this minimum ratio is a recommendation only. For the principles of this specification, see ISO 179-2:2020, Annex B^[5].



Кеу

- 1 test specimen
- 2 hemispherical striker tip
- 3 load cell (recommended position)
- 4 shaft

- 5 test specimen support
- 6 clamping ring (optional)
- 7 base
- 8 acoustical isolation (optional)

ue	Dimensions mm		
ecime type	Side length or diameter	60	140
Sp	Designation to ISO 20753	D12 and D22	-
	<i>D</i> ₂	40 ± 2	100 ± 5
di- ons	<i>D</i> ₃	60	140
mp nsid	<i>D</i> ₄	≥ 90	≥ 200
Cla	Н	12	12
	R	1	1

Figure 5 — Example of test device



Кеу

- 1 clamping ring (optional)
- 2 test specimen support

Figure 6 — Clamping device (schematic)

5.1.5 Clamping device (optional), consisting of two parts, a supporting ring and a clamping ring (see Figure 6), for annular test specimens. The recommended inside diameter of the clamping device is (40 ± 2) mm, alternatively (100 ± 5) mm. The clamp may work by shape or by application of force to the specimen. A clamping force of 3 kN is recommended for the latter (see NOTE).

NOTE Pneumatically and screw-operated clamps have been successfully employed. The results obtained for clamped and unclamped specimens are likely different (see <u>Annex C</u>).

5.2 Instruments for measuring force and deflection

5.2.1 Force measurement system, for measuring the force exerted on the test specimen. The striker may be equipped with strain gauges or a piezoelectric load transducer, which shall be placed close to the striker tip. Any other suitable method of force measurement is also acceptable. The measurement system shall be able to record forces with an accuracy equal to or within ± 2 % of the maximum impact force, F_M , which has occurred during the test.

The force measurement system shall be calibrated as set-up ready for measurement. Calibration should be performed statically (for example, by imposing known loads on the striker as described by reference [6]) or dynamically (see for example reference [4]). The range for which the force measurement system works within an accuracy of ± 2 % of the reading shall be indicated.

As the duration of the test is very short, only electronic load cells with a high natural frequency shall be used (see NOTE 1). The natural frequency f_n of the test device (striker and load cell) shall conform to the following condition:

$$f_{\rm n} \ge 6 \,\mathrm{kHz}$$
 (3)

For interpretation of complex force-deflection curves, even higher values of the natural frequency f_n can be necessary (see <u>Annex A</u>). For detecting the first damage depicted in <u>Figure A.2</u>, the natural frequency shall comply with the following condition (see NOTE 2):

$$f_{\rm n} \ge 5 / \Delta t_{\rm E}$$

where

- $f_{\rm n}$ is the natural frequency, expressed in kilohertz;
- $\Delta t_{\rm E}$ is the event time of the relevant detail of the force-deflection curve, expressed in milliseconds (see Figure A.2).

The natural frequency can be checked by studying the oscillations following brittle or splintering failure (see Figure 3).

For the bandwidth of the amplifier train (direct current or carrier frequency amplifier) the lower bandwidth limit is 0 Hz, and the upper bandwidth limit shall be at least 100 kHz, combined with a sampling frequency of at least 100 kHz (see NOTES 3 and 4).

NOTE 1 An example of such a measurement train is a piezoelectric load cell, mounted between the striker and the shaft (see Figure 5) and connected to a charge amplifier.

(4)

NOTE 2 If, for example, the increase in deflection $\Delta t_{\rm E} \cdot v_0$ during the event (see Figure A.1) is only 1 mm (10⁻³ m), at an impact velocity v_0 of 4,4 m s⁻¹, then the corresponding event time is $\Delta t_{\rm E} = [(10^{-3} \text{ m})/(4,4 \text{ m s}^{-1})] = 2 \times 10^{-4}$ s, resulting in the minimum natural frequency of $f_n \ge [5/(2 \times 10^{-4} \text{ s})] = 25 \text{ kHz}$.

NOTE 3 When testing very brittle products, elastic impact can cause resonant oscillations, thus making it difficult to interpret the force-deflection curve (see <u>Annex A</u>). In this case, it can be useful to carry out low-pass filtering on the recorded force-time diagram or parts of it, although the accuracy of the measurements is thereby reduced.

If post-test filtering is used, the type of filter and its essential characteristics are reported in the test report (see $\underline{10}$ l)).

NOTE 4 Vibration of the test specimen (see Figure A.3) and of the test device as well as uniform noise on the trace generates uncertainties of the measured maximum force (see <u>3.5</u>) but has virtually no effect on the puncture energy (see <u>3.9</u>).

5.2.2 Deflection measurement system, consisting of an electronic transducer for the determination of the deflection of the test specimen to yield a force-deflection diagram. This deflection measurement system shall be able to measure the relevant deflections to an accuracy equal to or within ± 2 %.

If the measurement chains for force and deflection measurement show a difference of their transit times, the time traces shall be synchronized by a time shift corresponding to this transit-time difference.

NOTE Such transit time differences generate a time offset in the force-deflection curve, which increases proportionally to the impact velocity.

With inertial-mass type machines, it is possible to measure a force-time diagram only and to calculate the deflection from double integration of the force signal in accordance with <u>8.2</u>.

5.3 Thickness gauge, as specified ISO 16012.

6 Test specimens

6.1 Shape and dimensions

The preferred test specimen is 60 mm \pm 2 mm square or 60 mm \pm 2 mm in diameter, with a thickness of 2,0 mm \pm 0,1 mm, and is used with the 40 mm diameter support ring. This corresponds to the specimen types D12 and D22 described by ISO 20753.

For thicknesses less than 1 mm, preferably use ISO 7765-2. Thicknesses greater than 4 mm fall outside the scope this document.

For testing brittle fibre-reinforced plastic composites and low failure strain plastics, a test specimen 140 mm \pm 2 mm square or 140 mm \pm 2 mm in diameter with a recommended thickness of 4,0 mm \pm 0,2 mm should be used with the 100 mm diameter support ring.

6.2 Preparation of test specimens

The test specimens shall be prepared in accordance with the relevant material specification. Where none exist, or when not otherwise specified, test specimens shall be prepared in accordance with ISO 293, ISO 294-3, ISO 295 or ISO 1268-1 as appropriate or machined from plates in accordance with ISO 2818 (see NOTE). The test specimens may also be prepared with a cutting or punching device, since there are no special requirements for the cut edges.

NOTE The preparation of test specimens 140 mm square or 140 mm in diameter by injection moulding is not yet covered by any International Standard.

Because the larger specimen is used primarily for fibre-reinforced plastic composites, it is recommended that they be made by machining from sheet material.

Test specimens taken from larger sheets or sections of sheet shall be taken from locations that are as uniformly distributed over the surface as possible. Non-homogeneous edge zones shall not be used. The thickness of these test specimens shall be the thickness of the sheet up to a thickness of 4 mm. If the sheet is more than 4 mm thick, the specimens shall be machined to 4 mm.

6.3 Non-homogeneous test specimens

In general, the test is conducted on either side of the specimen, selected at random. However, if there is a reason to believe that the results are dependent on which side of the specimen faces the striker, each side shall be tested separately. This especially holds for test specimens with textured surfaces, specimens lacquered on one side and specimens which are UV-aged. When assessing the influence of a one-sided treatment, the test specimen shall be impacted on the opposite side.

6.4 Checking the test specimens

The specimens shall be free of twist and warpage. Both surfaces shall be smooth and free of scratches, pits and sink marks to avoid notching effects.

The specimens shall be checked for conformity with these requirements by visual observation or by measuring with a thickness gauge.

Specimens showing any observable departure from one or more of these requirements shall be rejected.

6.5 Number of test specimens

If the test is conducted under constant conditions, at least five or, in cases of arbitration, 10 test specimens are required. If the measurements are to be made as a function of temperature, relative humidity or some other parameter, the number of test specimens may be reduced depending on the statistical scattering of the test results.

If a large number of test specimens are required, for example to determine the temperature dependence of the measured quantities, the test specimens shall be selected in accordance with statistical principles.

6.6 Conditioning of test specimens

The test specimens shall be conditioned as required by the relevant material specification or as agreed upon by the interested parties. Otherwise, the most appropriate conditions from ISO 291 shall be selected. For materials with impact properties that are non-sensitive to moisture, a control of the humidity is not necessary.

6.7 Pre-cooling

For testing at low temperatures, the test specimens are pre-cooled to the test temperature after conditioning and prior to the test. When the test equipment is at ambient temperature, the pre-cooling temperature may be set below the test temperature to compensate for a possible temperature increase during the transit of the test specimens to the test equipment.

NOTE A pre-cooling temperature set 2 K lower than the test temperature is often sufficient.

The duration of the pre-cooling procedure shall be such that it is sufficient for the test specimen to reach a temperature within 2 K of the set pre-cooling temperature.

NOTE A duration of 1 h is often regarded as sufficient. ISO 23529:2016, Annex A presents suitable precooling times for elastomers.

7 Procedure

7.1 Test atmosphere

7.1.1 General

Conduct the test in one of the standard atmospheres specified in ISO 291. Use class 2 unless otherwise agreed.

7.1.2 Ambient temperature testing

If a standard atmosphere from ISO 291 was used for conditioning, conduct the test in the same atmosphere. If not, ensure that the transit time $t_{\rm T}$ (see NOTE) is short enough to prevent changes in the mechanical behaviour (state of material) of the test specimen caused by changes in the temperature and, if relevant for the material, the moisture content of the specimen. For dry polyamides, for instance, a transit time of up to 30 min has been found not to markedly affect the impact behaviour when testing in an atmosphere of 23 °C and 50 % R.H.

NOTE The transit time $t_{\rm T}$ is the total time from the removal of the specimen from the pre-cooling environment until the specimen is impacted.

7.1.3 Low temperature testing

When test specimens are pre-cooled to the test temperature and the test equipment is at room temperature, a transit time t_T (see note to 7.1.2) short enough to prevent significant changes in the temperature of the test specimen prior to impact is required (i.e. less than 5 s).

NOTE Differences in humidity between the test specimens conditioning atmosphere and the test atmosphere can be critical.

7.2 Measurement of thickness

For each test specimen, measure the thickness in accordance with ISO 16012 to the nearest 0,02 mm at three points which are equidistant to one another on a circle with a radius of 10 mm centred to the centre of the specimen. Record the lowest value of the measured thickness (see NOTE). If the thickness of any specimen differs by more than 5 % from the thickness of the specimens from that sample, discard that specimen and replace it with another specimen.

NOTE When using injection-moulded specimens, it is not necessary to measure the dimensions of each specimen. It is sufficient to measure one specimen from each set.

When using multiple-cavity moulds, measure the thickness of the specimens from each cavity. If the difference in specimen thickness between mould cavities is greater than 5 %, the specimens from each cavity shall be treated as different batches.

7.3 Clamping the test specimen

The default condition for this test is that the specimen is clamped.

If the specimen is clamped, however, take care to ensure that the clamping force does not induce bending or torsional forces in the specimen.

7.4 Lubrication

Lubricate the tip of the striker with oil or grease before each test. The viscosity η of the lubricant shall be in the range 10 cP < η < 10⁴ cP (10⁻² Pa·s < η < 10 Pa·s) (see <u>Annex B</u>)

7.5 Puncture test procedure

Place the test specimen on the specimen supporting ring (5.1.3) and clamping device (5.1.5) as appropriate.

Conduct the puncture test with the impact velocity specified in <u>5.1.1</u>. Ensure that the velocity does not change during the puncture process by more than 20 % by checking the deflection-time trace or by using Formulae (1) and (2) with the energy E^* equal to E_p .

8 Calculations

8.1 Expression of results

Take the force-time curve or, where directly measured, the force-deflection curve as the test result (see also <u>3.9</u>, Note 3 to entry). Other results shall be calculated employing these data.

For the purposes of routine characterization and in the absence of other conditions described in the International Standard for the material concerned, the values of the following properties shall be taken as results of the test:

- a) $l_{\rm M}$ is the deflection at maximum force (see <u>3.6</u>), expressed in mm;
- b) $E_{\rm M}$ is the energy to maximum force (see <u>3.7</u>), expressed in J;
- c) $F_{\rm M}$ is the maximum force (see <u>3.5</u>), expressed in N;
- d) $l_{\rm P}$ is the puncture deflection (see <u>3.8</u>), expressed in mm;
- e) $E_{\rm p}$ is the puncture energy (see <u>3.9</u>), expressed in J.

Additionally, the type of impact failure as defined in 3.10 and by Figures 1 to 4 should be reported. For failure types YS and YU, ensure that frictional forces do not affect the force-deflection diagram at large deflections. For complex behaviour see Annex A.

8.2 Calculation of deflection

If the test results are in the form of a force-deflection curve, the maximum force F_M , the deflection at maximum force l_M and the puncture deflection l_P can be read directly from the graph. The energy to maximum force E_M and the puncture energy E_P (see Figures 1 to 4) can be determined by measuring the area under the force-deflection curve, using a planimeter, computer analysis or other suitable means.

For falling mass type energy carriers (see 5.1.1.2) that show nominally no frictional loss during impact, the deflection of the test specimen cannot be measured directly by a displacement measuring system. In this case, it shall be calculated from the force-time trace using Formula (5).

$$l(t) = v_0 t - \frac{1}{m_c} \int_0^t \left[\int_0^t F(t) dt_1 \right] d(t) + \frac{1}{2} g t^2$$
(5)

where

- v_0 is the impact velocity (see <u>3.1</u>), expressed in metres per second;
- *t* is the time after impact at which the deflection is to be calculated, expressed in seconds;
- F(t) is the force measured at any time after the impact, expressed in newtons;
- l(t) is the deflection (see <u>3.3</u>), expressed in meters;
- $m_{\rm C}$ is the falling mass of the energy carrier, expressed in kilograms;

g is the local acceleration due to gravity, expressed in metres per second squared.

Since the last term of <u>Formula (5)</u> is only valid for an energy carrier moving vertically, its relative contribution increases with decreasing impact velocity (drop height of the striker).

8.3 Calculation of energy

Once the force and deflection are known for identical times during impact, the energy expended up to specific times t_j shall be calculated by determining the area under the force-deflection curve according to Formula (6) (see NOTE 1).

$$E_{j} = \int_{0}^{l_{j}} F(l) dl \tag{6}$$

where

- *F*(*l*) is the force at the deflection l, expressed in newtons;
- *l* is the deflection, expressed in metres;
- j is a subscript denoting one of the following points:
 - M = maximum
 - P = puncture;
- *E* is the energy, expressed in joules.

NOTE 1 The energy to maximum force and the puncture energy can be determined utilizing electronic integration.

In the case of frictionless energy carriers, impacting horizontally, the energy can also be calculated without developing the deflection/time trace, using the Formulae (7) and (8) (see NOTE 2).

$$E_{j} = E_{ja} \left(1 - \frac{E_{ja}}{4E_{c}} \right)$$

$$E_{ja} = v_0 \int_0^{t_j} F(t_1) dt_1$$
(8)

where

- E_{ja} is the approximate value of the energy, calculated assuming a constant velocity v_0 , expressed in joules;
- E_c is the energy of the energy carrier just before the impact, expressed in joules;
- $F(t_1)$ is the force at the time t_1 , expressed in newtons.

NOTE 2 Formula (7) is based on the conservation of energy and momentum, omitting the influence of gravity. The second term within brackets is less than 5 % if the ratio E^*/E_c of the maximum energy to be measured to the capacity of the energy carrier is less than 0,2.

8.4 Statistical parameters

Calculate the arithmetic mean, the standard deviation and the coefficient of variation of the properties named in <u>8.1</u> for each test series (see ISO 2602).

8.5 Significant figures

Report all calculated mean values to three significant figures.

9 Precision

An example of precision data for this test method is shown in <u>Annex G</u>.

10 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 6603-2:2023;
- b) the test parameters, identified as follows:
 - the support ring diameter 40 mm (or 100 mm),
 - the striker diameter 20 mm (or 10 mm),
 - the clamping situation of the specimen, clamped C, or unclamped U,
 - the impact velocity 4,4 m/s (or other),
 - e.g. "Instrumented puncture test ISO 6603-2/40/20/C/4,4";
- c) the type, identification mark, origin, date of receipt and other pertinent data concerning the test material, such as coated, textured and orientation of texture;
- d) the shape and dimensions of the test specimens, or the specimen type, if in accordance with ISO 20753;
- e) the method of preparation of the test specimens;
- f) the lowest thickness of the test specimens, measured in accordance with <u>7.2</u>;
- g) the test conditions and, if applicable, the conditioning and pre-cooling procedure;
- h) the number of test specimens tested;
- i) the appearance of the test specimens after the test (optional);
- j) the impact-failure criterion that was agreed upon, if different from that given in <u>3.10</u>;
- k) the natural frequency of the force-measuring device;
- l) the type and essential characteristics of post-test filtering, if used;
- m) the individual test results, arithmetic mean, standard deviation or coefficient of variation and the 95 % confidence intervals of these mean values in accordance with ISO 2602 of the following properties, if required:
 - the maximum force F_{M} , expressed in newtons;
 - the deflection at maximum force l_{M} , expressed in millimetres;
 - the energy to maximum force E_{M} , expressed in joules;
 - the puncture energy $E_{\rm p}$, expressed in joules;
 - the puncture deflection $l_{\rm p}$, expressed in millimetres.
- n) the type of failure (see <u>3.10</u>);

- o) the force-deflection or force-time curves;
- p) the date of the test.

Annex A

(informative)

Interpretation of complex force-deflection curves

In many impact experiments, the force-deflection diagram is more complicated than those shown in Figures 1 to $\frac{4}{2}$. In such cases, a point of damage D cannot be derived in any simple way from the force-deflection diagram using a standard procedure.

However, by means of an accurate comparison of the force-deflection diagram with the specimen tested, in many cases a reliable statement about the agreed point of damage can be made.

Practically, an impact experiment can be conducted with a lower energy (falling height) using inertial mass systems, respectively lower testing speed using hydraulically driven systems. In the first case, the available energy shall be selected slightly larger than the assumed puncture energy.

This method is especially recommended for the testing of brittle or textile-reinforced materials. In these cases, a dip in the rising part of the force-deflection diagram is found indicating first damage, D (see Figure A.1).

Although for brittle and fibre-filled materials the maximum force usually corresponds to the force of crack initiation, very often a second peak occurs due to the formation of the crack necessary for the penetration of the striker (see Figures A.1 and A.2).

Many peaks in the force-deflection diagram can appear due to resonance (see Figure A.3). The interpretation of such a diagram is very difficult, even when the condition given in 5.2.1 on the natural frequency of the test device is met.

A visual assessment of the broken specimen is then the only way of describing the fracture behaviour under impact.



- X deflection
- Y force

NOTE First damage (D) followed by puncture (P), where Δt_E is the event time of the first damage in the force-time trace and v_0 the impact velocity.

Figure A.1 — Schematic force-deflection diagram for brittle or textile-fibre reinforced material indicating first damage followed by puncture



Кеу

- X deflection
- Y force

Figure A.2 — Schematic force-deflection diagram for a brittle or textile-fiber reinforced material



X deflection

Y force

Figure A.3 — Schematic force-deflection diagram for splintering material, superposed by strong resonance of the test specimen

Annex B

(informative)

Friction between striker and specimen

As a result of biaxial symmetric stress, the failure of the specimen in a puncture test is expected to occur at the point of maximum theoretical stress, i.e. at the centre of the specimen. However, a frequent observation is a circular crack and a subsequent punching out of a round cap. Evidently, this effect results from a drop in the amount of stress at the top point due to friction. The maximum stress and consequently the locus of failure shifts to the circle of contact between the striker and the specimen (see Figure B.1). The part of the specimen volume, therefore, which stores and absorbs energy during the test, strongly depends on friction. Additionally, other disadvantages can occur due to friction.

- Due to the action of an unknown amount of friction, the forces appearing in a puncture test are
 increased in an uncontrolled way.
- For some materials a friction-caused abrasion of the polymer can be observed. The abraded material clings onto the striker tip together with other deposits resulting, for example, from additives like demoulding agents and external lubricants. As a result of this deposit, a distinct increase in scatter occurs which can only be reduced by cleaning the tip carefully before each test (see Figure B.1).
- The type of cooling has a strong influence on puncture ductility. When cold specimens are tested at room temperature, a thin film of water or ice from the atmospheric humidity condenses on the specimen surface and acts as a lubricant. Therefore, an apparent step in the temperature-dependent ductility occurs at about 0 °C (see Figure B.2).
- The results of puncture can be influenced by the striker material, its surface roughness and that of the specimen tested.
- Lubricating or greasing the striker overcomes these disadvantages. The failure of the specimens occurs at its centre, as expected. By concentrating the plastic deformation at the centre of the specimen instead of spreading it over a large, undefined portion of the specimen volume, scattering is reduced and comparable data can be obtained. The values obtained with a lubricated striker are unequivocal lower limits of the tested mechanical properties of the material.
- Tests at a high standard test speed of 4,4 m/s using lubricants in the viscosity range of 0,01 Pa·s<ŋ<10 Pa·s have shown that the type of lubricant is not relevant. At test speeds lower than 1 m/s, however, low-viscosity lubricants can be squeezed out of the contact area and can result in diverging values. At test speeds lower than 10⁻² m/s, results tend to be similar to those of specimens tested without lubrication. Sufficient lubrication, however, generally can be controlled by checking the locus of failure (see Figure B.1).



Figure B.1 — Force-deflection curves and the appearance of test specimens with tough failing, tested with and without lubrication of the striker, for example using Vaseline



Figure B.2 — Puncture energy as a function of temperature with and without specimen lubrication

Annex C

(informative)

Clamping of specimens

The method of supporting and holding the specimen can have some influence on the puncture behaviour of the given material. Testing unclamped specimens in the case of an unlubricated striker can lead to deformations of the supported outer region of the specimens, making the test specimen change from an initially planar shape to a conical, wavy shape. This additional deformation energy can cause the puncture toughness to be apparently higher than that of clamped specimens which have been prevented from buckling. Until now, however, only minor effects have been found which have been reduced by lubricating the striker. Furthermore, higher vibration amplitudes can be shown for unclamped test specimens.

A clamping device cannot fully prevent the test specimen from radial slippage. However, it is important to prevent the supported part of the test specimen from buckling. This can be achieved by clamping the test specimen with sufficient force or by clamping by shape (small clamping force, but rigid construction). Clamping by shape and by friction have not shown significant differences in the puncture behaviour.

Annex D

(informative)

Tough/brittle transitions

Tough/brittle transitions are often encountered in a series of tests carried out at decreasing temperature. At such transitions, the puncture energy, for example, decreases and/or the type of failure changes. The cause of these transitions are molecular relaxation processes which become effective only above a certain temperature and which increase the absorption of the impact energy.

Test time plays a role similar to that of temperature, i.e. if the test time is shortened by increasing the impact velocity, the transition temperature is shifted to higher values. The relationship between time and temperature is determined by the temperature dependence of molecular relaxation times, which is approximated by the Arrhenius equation shown as Formula (D.1):

$$t = t_0 * \exp(\frac{E_A}{kT}) \tag{D.1}$$

where

- *t* is the relaxation time or test time;
- *T* is the absolute temperature of the tough/brittle transition;
- $E_{\rm A}$ is the apparent activation energy.

Within a transition region, a wide scatter of results often is observed, for example the failure of the test specimens being brittle or tough at the same temperature. In high-density polyethylene, for example, such a transition region is found in the temperature range between -140 °C and -105 °C, depending on the relative molecular mass and degree of crystallinity.

Tough/brittle transitions can be recognized by means of the force-deflection diagram or from the appearance of the damaged specimens (see Figures 1 to 4).

For plastics exhibiting a tough/brittle transition, the performance and evaluation of impact tests are subject to certain limitations, since the specimens of a single test series are to be assigned to two different parent populations, namely one exhibiting tough behaviour and one exhibiting brittle behaviour. In such cases, the means and variances are not statistically defined over the entire range of measurements. Nevertheless, it is helpful to employ the mean and standard deviation calculated from the individual measurements to characterize the behaviour of the material.

Where there is a sufficient number of measurements for both parent populations, the characteristic quantities can be calculated separately for the brittle and the tough test specimens. If necessary, the choice of assigning measurements to one of the two parent populations should be decided by use of the statistical procedure normally employed for this purpose.

Annex E

(informative)

Influence of specimen thickness

The forces obtained in a puncture test depend on the thickness of the test specimen. By increasing the thickness of the test specimen the resulting forces are greater than proportional. This is due to the different deformation processes occurring in a puncture test, i.e. small deflections cause biaxial flexural loads to turn into biaxial tension with increasing deflection.

In the region of linear elastic material behaviour, the relationship between the thickness and the resulting force can be described as $F(l) \sim w^n$. The exponent *n* changes during the test from n = 1 (flexural deformation) to n = 3 (tension) in a way depending on the material and the test conditions.^{[2],[3]} Therefore, the normalization of a complete force-deflection curve to a given thickness is impossible. For selected properties, however, the thickness dependence can be fitted [see Formulae (E.1) and (E.2)]. As an example, for polycarbonate sheets the maximum force and the puncture energy depending on the thickness w are shown in Figure E.1 in a double-logarithmic plot.

$$F_{\rm M} \sim w^n \tag{E.1}$$

$$E_{\rm p} \sim w^m \tag{E.2}$$

<u>Table E.1</u> shows the typical range of the exponents n and m for the maximum force and the puncture energy found for a series of thermoplastics and the relative deviations caused by a relative scatter of the thickness of 5 %.

Table E.1 — Exponents, n or m, for fitting the thickness dependence of maximum force F_M and puncture energy E_P and the effect of the scatter of thickness on that of the properties

Ducantar	Exponent	Effect of the scatter			
Property	<i>n</i> or <i>m</i>	of thickness $\Delta w/w$ of ±5 % on that of the property			
F _M	1,0 to 1,4	±5 % to ±7 %			
E _P	1,2 to 1,8	±6,5 % to ±9 %			

As can be seen, even small changes in thickness of 5 %, which are admissible according to this document, result in distinct deviations of maximum force and puncture energy.

Plotting the test results according to <u>Formulae (E.1)</u> and <u>(E.2)</u> (see for example <u>Figure E.1</u>) can give interpolation facilities and may show the source of scatter of data. The consequence is an increased comparability of results obtained at specimens with varying thickness, particularly testing specimens taken from compression-molded plates or from parts.



Кеу

- X specimen thickness (w), mm
- Y1 maximum force (F_M), N
- Y2 puncture energy (E_p), J

Figure E.1 — Maximum force a) and puncture energy b) of a polycarbonate with respect to specimen thickness *w*

Annex F (informative)

Guidance for the classification of the type of failure

F.1 General

The classification of the type of failure is a matter of the shape of the force-time or the force-deflection curve and the appraisal of the broken specimen. The following real examples are intended to give further guidance for a correct classification in accordance with <u>Clause 3</u>.

F.2 Examples

Force-deflection diagram	Broken specimen	Type of failure	
PP natural, not reinforced Y 2 500 2 000 1 500 1 000 500 0 2 4 6 8 X Key X deflection [mm] V force [N]		YD yielding, followed by deep drawing	
The curve shows a clear zero-slope at the maximum force, followed by deep drawing deformation, shown by a slow and steady decrease of the force.			

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Force-deflection diagram	Broken specimen	Type of failure	
PP-TPO natural, not reinforced Y 2 000 1 500 500 0 10 20 30 X		YD yielding, followed by deep drawing	
KeyXdeflection [mm]Yforce [N]Same curve shape as before, but at lower forceand higher deformation.	Fracture started well nearby the tip of the striker, but the shape of the deformation looks slightly different to the first example.		
ABS natural, not reinforced Y 3 000 2 000 1 000 0 5 10 15 20 X Key X deflection [mm] Y force [N] The curve shows only few yielding. There is unstable cracking after the maximum force, shown by a significant sudden drop of the force.	The failure started at the striker tip. The crack propagated to the edge of the clamped area. The appearance is mainly brittle. Ductile deformations, appearing as whitening in colour are significant for vielding.	YU yielding followed by unstable cracking	



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Table F.1 — Examples for the classification of different types of failure

Annex G (informative)

Precision data

G.1 General

<u>Table G.1</u> is based on interlaboratory tests performed in 2010 by the Kunststoffinstitut Lüdenscheid, Germany, in accordance with ISO/IEC 17043 and ISO 13528. The test results have been evaluated in accordance with ISO 5725-2. For each material, all test specimen were prepared by one single source. Each laboratory was requested to test 3 specimens for each material and temperature.

The reported results were submitted to 3 different outlier tests:

- Grubbs's test as described by ISO 5725-2, based on the reported average values, determining the compatibility of the reported results from one lab to all other results reported
- Cochran's C test as described by ISO 5725-2, comparing the standard deviations
- Z-score as described by ISO 5725-2, a measure of how many standard deviations below or above the
 population mean the average of a single lab result is.

Results which failed to pass at least one of the outlier tests have been rejected.

The tables show the following statistical results:

- the within-laboratory standard deviation, s_r as absolute value and as percentage of the average
- the between-laboratory standard deviation, s_R as absolute value and as percentage of the average

NOTE The data in <u>Table G.1</u> are not intended to be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and might not be representative of other lots, conditions, materials or laboratories.

G.2 Concept of *r* and *R*

If s_r and s_R have been calculated from a large enough body of data, and for test results that were from one test determination, then:

- For the **repeatability limit**, *r* (comparing two test results for the same material, obtained by the same operator using the same equipment on the same day), the two test results should be judged not equivalent if they differ by more than the *r*-value for that material. Repeatability limit, $r = 2,83 s_r$;
- For the **reproducibility limit**, *R* (comparing two results for the same material, obtained by different operators using different equipment on different days), the two test results should be judged not equivalent if they differ by more than the *R*-value for that material. Reproducibility limit, *R* = 2,83 s_R.

Any judgment in accordance with the above would have an approximately 95 % (0,95) probability of being correct.

Table G.1 — Precision data for different types of results, materials and temperatures

Maximum Force, F_M

Maximum Fore	с, т _М	· · · · · · · · · · · · · · · · · · ·				,	
Material	Temperature °C	N° of labs	Average in N	s _r in N	s _R in N	rel s _r	rel s _R
POM (white)	Ambient	8	3 780	83,5	89,8	2,2 %	2,4 %
PP-GF (black)	Ambient	8	1 210	45,0	45,2	3,7 %	3,7 %
POM (white)	-30	9	4 020	429	588	10,7 %	14,6 %
PP-GF (black)	-30	7	1 300	39,1	75,0	3,0 %	5,8 %
Deflection at m	naximum force, I _M						
Material	Temperature °C	N° of labs	Average in mm	s _r in mm	s _R in mm	rel s _r	rel s _R
POM (white)	Ambient	10	13,0	0,48	0,51	3,7 %	3,9 %
PP-GF (black)	Ambient	8	4,73	0,48	0,53	10,1 %	11,2 %
POM (white)	-30	9	8,39	0,83	1,30	9,9 %	15,5 %
PP-GF (black)	-30	9	5,11	0,26	0,30	5,1 %	5,9 %
Energy to maxi	imum force, E _M						
Material	Temperature °C	N° of labs	Average in J	s _r in J	s _R in J	rel s _r	rel s _R
POM (white)	ambient	10	24,2	2,00	2,17	8,3 %	9,0 %
PP-GF (black)	ambient	10	3,21	0,46	0,56	14,3 %	17,4 %
POM (white)	-30	9	14,5	2,88	3,28	19,9 %	22,7 %
PP-GF (black)	-30	9	4,06	0,33	0,56	8,1 %	13,8 %
Puncture defle	Puncture deflection, <i>l</i> _P						
Material	Temperature °C	N° of labs	Average in mm	s _r in mm	s _R in mm	rel s _r	rel s _R
POM (white)	ambient	10	14,1	0,75	0,76	5,3 %	5,4 %
PP-GF (black)	ambient	9	8,81	0,38	0,51	4,3 %	5,8 %
POM (white)	-30	8	8,10	0,77	0,97	9,5 %	12,0 %
PP-GF (black)	-30	8	8,70	0,59	0,64	6,8 %	7,4 %
Puncture Energy, <i>E</i> _P							
Material	Temperature °C	N° of labs	Average in J	s _r in J	s _R in J	rel s _r	rel s _R
POM (white)	ambient	10	28,0	2,81	2,97	10,0 %	10,6 %
PP-GF (black)	ambient	9	7,13	0,38	0,39	5,3 %	5,5 %
POM (white)	-30	8	14,2	1,97	2,52	13,9 %	17,8 %
PP-GF (black)	-30	9	7,48	0,79	0,83	10,6 %	11,1 %
NOTE The variation of n° of labs even for the same material and condition variates is because single laboratories reported							

NOTE The variation of n° of labs even for the same material and condition variates is because single laboratories reported results failing the Z-score outlier test. These results have been rejected as explained in $\underline{G.1}$

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