INTERNATIONAL STANDARD

ISO 22088-4

First edition
2006-08-15

Plastics — Determination of resistance to environmental stress cracking (ESC) —

Part 4: Ball or pin impression method

Plastiques — Détermination de la fissuration sous contrainte dans un environnement donné (ESC) —

Partie 4: Méthode par enfoncement de billes ou de goupilles

Reference number
ISO 22088-4:2006(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.

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 22088-4 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

It cancels and replaces ISO 4600:1992, which has been technically revised.

ISO 22088 consists of the following parts, under the general title *Plastics — Determination of resistance to environmental stress cracking (ESC)*:

— *Part 1: General guidance*

— *Part 2: Constant tensile load method* (replacement of ISO 6252:1992)

— *Part 3: Bent strip method* (replacement of ISO 4599:1986)

— *Part 4: Ball or pin impression method* (replacement of ISO 4600:1992)

— *Part 5: Constant tensile deformation method* (new test method)

— *Part 6: Slow strain rate method* (new test method)

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Plastics — Determination of resistance to environmental stress cracking (ESC) —

Part 4: Ball or pin impression method

1 Scope

1.1 This part of ISO 22088 specifies a ball or pin impression method for the determination of the environmental stress cracking (ESC) behaviour of plastics by means of a constant-strain test.

1.2 The method is applicable to finished products and to test specimens prepared by moulding and/or machining, and can be used for the assessment of the ESC behaviour of a plastic product or material exposed to different environments, as well as for the determination of the ESC behaviour of different plastics materials exposed to a specific environment.

NOTE Alternative methods for the determination of environmental stress cracking by means of a constant-strain test are specified in ISO 22088-3 and ISO 22088-5. A method for the determination of environmental stress cracking by means of a constant-stress test is specified in ISO 22088-2.

1.3 The ball and pin impression methods are both quick and sensitive procedures for assessing the ESC behaviour of plastics. The methods are well suited for amorphous plastics. They are less appropriate for materials displaying a pronounced tendency for creep and/or stress relaxation, i.e. for semi-crystalline materials. If semi-crystalline materials are tested, pins are more appropriate than balls.

1.4 The ball impression method is useful for assessing the principal ESC behaviour of the material/chemical combination under consideration. It is less influenced by the near-surface orientation state of the specimens than the pin impression method and the methods in the other parts of this International Standard, where the chemical attacks only the original surface of the material. This, depending on the manner of specimen preparation, may show a considerable degree of orientation.

1.5 The pin impression method is useful for testing specimens of small thickness and finished parts.

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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 178, *Plastics — Determination of flexural properties*

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

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

ISO 294-1, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens*

ISO 527-1, *Plastics — Determination of tensile properties — Part 1: General principles*

ISO 527-2, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics*

ISO 2557-1, *Plastics — Amorphous thermoplastics — Preparation of test specimens with a specified maximum reversion — Part 1: Bars*

ISO 2818, *Plastics — Preparation of test specimens by machining*

ISO 3167, *Plastics — Multipurpose test specimens*

ISO 3290, *Rolling bearings — Balls — Dimensions and tolerances*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

3 Terms and definitions

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

3.1
oversize
d_d
difference between the diameter of an impressed ball or pin (d_b) and the diameter of the drilled and reamed hole (d_h) in the test specimen:

$$d_d = d_b - d_h \quad (1)$$

3.2
deformation step
determination made at a defined oversize

3.3
deformation step zero
determination made using test specimens that are drilled and reamed only, i.e. without impressing a ball or pin

3.4
deformation series
number of successive deformation steps beginning with deformation step zero

NOTE Normally, a deformation series consists of seven deformation steps of increasing severity.

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3.5
failure limit
oversize in a deformation series that produces failure, as specified in terms of the following failure criteria:

a) for type A test specimens (test specimens taken from products), as visible cracks, observable by means of a lens of magnification ×5;

b) for type B test specimens (moulded or machined test specimens), by the following criteria (see 9.2 and Figure 4):

   1) a 5 % reduction in the maximum tensile force measured at deformation step zero (criterion B1 in Figure 1),

   2) a 5 % reduction in the maximum flexural force measured at deformation step zero (criterion B2 in Figure 1),

   3) a 20 % reduction in the tensile elongation at rupture measured at deformation step zero (criterion B3 in Figure 1).

NOTE 1 If there is no rupture immediately after application of the maximum tensile force, the tensile elongation at 50 % of the preceding maximum tensile force (see Figure 1) may be measured. Failure is then defined by a 20 % reduction in the value at deformation step zero (criterion B4).

NOTE 2 It is sufficient to measure the elongation at break between the grips.

NOTE 3 If the value of the tensile stress is required, refer the force to the smallest cross-sectional area of the specimen at the location of the hole. Calculate the tensile stress in accordance with Equation (2):

$$\sigma = \frac{F}{h \cdot (w - d_{\mathrm{h}})} \tag{2}$$

where

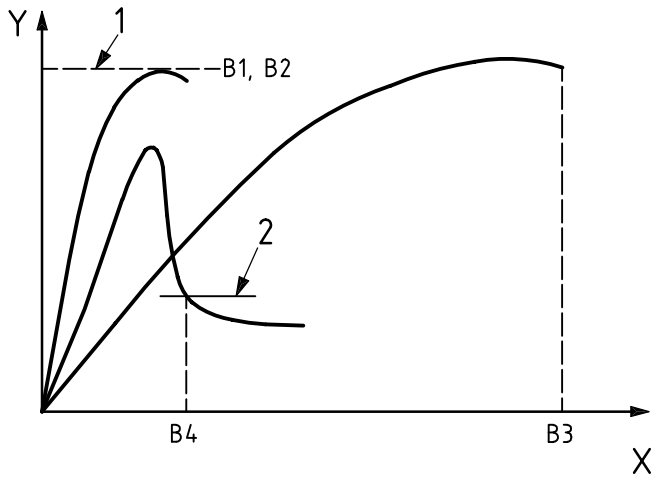
σ is the tensile stress, in MPa or $N \cdot mm^{-2}$;

F is the tensile force, in N;

h is the thickness of the specimen, in mm;

d_{h} is the diameter of the hole, after reaming, in mm;

w is the width of the specimen, in mm.



Key

X	elongation	B1	5 % reduction in the maximum tensile force
Y	stress	B2	5 % reduction in the maximum flexural force
1	maximum	B3	20 % reduction in the tensile elongation at rupture
2	0,5 × preceding max. tensile force	B4	tensile elongation at 50 % of the preceding maximum tensile force

Figure 1 — Failure criteria for type B test specimens

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3.6
relative stress-cracking factor
ratio of the failure limit in the test environment to that in a reference environment, for example air, measured at the same test temperature after the same test time

4 Principle

A constant strain, produced by impressed balls or pins in a test specimen in a test environment, often generates micro-cracks that may, in time, develop into visible cracks. To shorten the time for the test, failure may be accelerated by subsequent mechanical testing. If products cannot be assessed by mechanical tests, visual examination for cracks around the balls or pins may be undertaken.

A hole of specified diameter is drilled in a test specimen, an oversize ball or pin is inserted into the hole and the specimen is brought into contact with a chemical medium. This procedure is repeated using balls or pins of progressively greater diameter. After a specified time, the effect of the interaction is determined by visual examination (type A test specimens) or by the determination of the tensile or flexural properties (type B test specimens). A parallel series of tests may be performed in which the test specimens are exposed to air, and the comparative behaviour determined.

NOTE Pins are suitable for a single series of test specimens or articles of thickness greater than 1 mm. The deformation of the test specimen is the same along the whole length of the hole. Balls are suitable for thicknesses greater than 2 mm. The preferred thickness is 4 mm.

5 Apparatus

5.1 Drilling machine, operating at a suitable frequency of rotation, for example at 1 000 min^{-1}.

5.2 Drills, of diameter (2,8 $\pm$ 0,1) mm.

5.3 Reamer, suitable for finishing a hole of diameter (3,00 $\pm$ 0,05) mm.

NOTE A 3H7 reamer (3,004 mm to 3,008 mm) is suitable.

5.4 Plug gauges, or other suitable devices, for measuring the diameters of the reamed holes to within 0,005 mm.

5.5 Micrometer, for determining the diameters of the pins with an accuracy of 0,001 mm.

5.6 Polished steel balls or **pins**, having tolerances of $\pm$ 0,001 mm on diameters up to 4 mm and $\pm$ 0,01 mm on diameters greater than 4 mm.

NOTE If steel is attacked in the test environment, other suitable hard materials, for example glass, may be used for the balls or pins.

The use of the ranges of diameters given in Table 1 is recommended.

5.6.1 Balls, conforming to ISO 3290 grade G20 for diameters up to 4 mm and grade G200 for diameters greater than 4 mm.

5.6.2 Pins, free of roughness or sharp edges, having a parallel-sided part 10 mm to 50 mm long and a taper (1:5) at one end to reduce the entry diameter to 2,5 mm (see Figure 2). The surface roughness of the pins shall be equal, preferably with $Ra < 0{,}02$ µm (see ISO 4287).

NOTE A longer parallel-sided part of the pin will allow several test specimens to be tested with the same pin.

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5.7 Jig, for drilling and reaming the holes (a typical fixture is shown in Figure 3).

5.8 Apparatus for pressing the balls or pins into the hole.

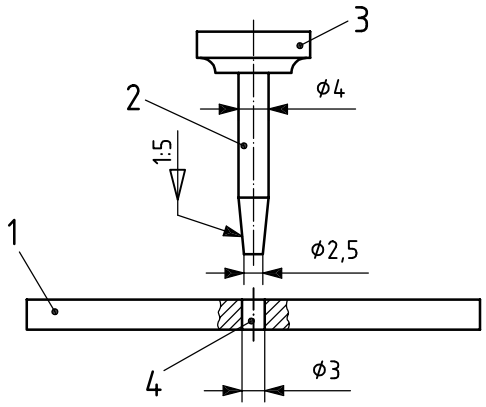
The spindle of the drilling machine or the tensile-testing machine itself may be used.

Table 1 — Recommended ranges of diameter for balls and pins

Dimensions in millimetres

Diameter	Increment
2,98 to 3,20	0,01
3,20 to 3,50	0,05
3,50 to 4,00	0,10
4,0 to 6,0	0,50

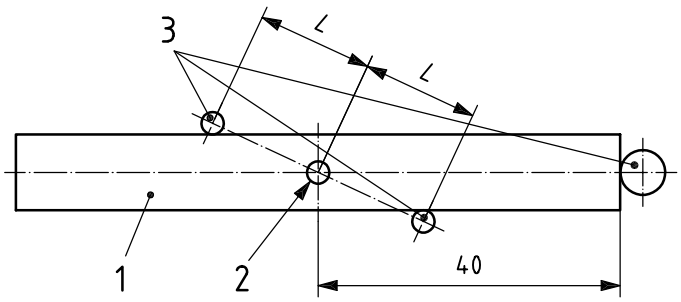
Dimensions in millimetres



Key
1 test specimen
2 pin
3 presser foot of test machine
4 drill hole

Figure 2 — Example of pin construction and insertion of the pin

Dimensions in millimetres



Key
1 test specimen
2 hole
3 holding pins

Figure 3 — Typical fixture for drilling holes in test specimens

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5.9 Vessels, for immersion of the specimens. Vessels shall be made of suitable material, which is not attacked by the test environment and will not interact with the immersion liquid.

5.10 Clock.

5.11 Flexural- or **tensile-testing machine** (see ISO 178 or ISO 527-1, respectively), for the determination of flexural or tensile properties.

6 Test specimens

6.1 Shape

6.1.1 General

In general, use test specimens of the shape and method of preparation specified in the International Standard appropriate to the material or product concerned.

If the relevant International Standard contains no such specifications, test specimens of the following shape shall be used.

6.1.2 Type A test specimen

Use the product or part of it as the test specimen.

6.1.3 Type B test specimen

Use moulded or machined test specimens conforming to ISO 293, ISO 294-1 or ISO 2818, as applicable.

Test specimens shall not be machined on the faces where the holes will be drilled. If test specimen dimensions are not specified, for flexural testing use a bar of dimensions 80 mm × 10 mm × 4 mm, as specified in ISO 178, and for tensile testing use the appropriate test specimen specified in ISO 527-1 and ISO 527-2. Attention is drawn to the multipurpose specimen specified in ISO 3167.

NOTE 1 Flexural testing has the following disadvantages:

In three-point flexure, the load is introduced directly at the hole. This may interfere with balls in the specimen.

Ductile specimens do not always break in flexure. It may therefore be impossible to determine the relative stress-cracking factor due to the fact that the deformation step zero value is missing.

NOTE 2 Tensile testing may also be performed using specimens measuring 80 mm × 10 mm × 4 mm, prepared directly or cut from the central part of the multipurpose test specimen. This allows smaller vessels and a smaller amount of immersion liquid to be used.

6.2 State

For tests which are intended to be comparable, the test specimens shall be in the same state. Attention is drawn to ISO 2557-1 for the determination of level of shrinkage and to ISO 294-1 for the state of the specimens. If finished articles are tested, the holes shall be drilled in the same area, or in areas agreed upon by the interested parties, especially if critical regions, such as weld lines, are to be examined.

The level of shrinkage of the test specimens, whether compression moulded, injection moulded or machined from sheet, shall be determined on five test specimens before they are drilled and reamed.

When evaluating moulding materials made of crystalline polymers, such as polyethylene and polypropylene, the amount of crystallinity shall be controlled by following the instructions given in the standard relevant to the material being tested, or as agreed between the interested parties.

NOTE The numerical value of the failure limit depends upon the method of determination and the distance between the edge of each hole and the side of the specimen. The value decreases as this distance decreases.

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6.3 Number of test specimens

6.3.1 General considerations

The number of test specimens required depends upon the duration of the test, i.e. short (see 8.4.2) or long (see 8.4.3), and the method used. Three deformation steps shall lie on either side of the expected approximate failure limit. When a long exposure is carried out with evaluation at intermediate intervals, additional specimens will be necessary.

6.3.2 Short-duration test (5 min to 24 h in the test environment) (see 8.4.2)

6.3.2.1 Selection of chemical medium

In view of the handling operations required for immersing the specimens in the chemical medium and removing them again, it has been found that 5 min is the shortest practical length of time for immersion. If it is possible to choose between different chemical media, it is recommended that one be selected which gives a measurable amount of stress cracking in not less than 1 h.

6.3.2.2 Type A test specimen

Three complete deformation-step series shall be used for testing. The required number of test specimens depends on the number of holes that can be drilled in the homogeneous region of each specimen.

6.3.2.3 Type B test specimen

Five test specimens shall be used for each deformation step.

6.3.3 Long-duration test

The number of test specimens depends upon the test conditions (see 8.4.3).

7 Conditioning and test conditions

7.1 Conditioning

Unless otherwise agreed between the interested parties (e.g. for polyamides or ABS), the test specimens shall be conditioned for at least 24 h at (23 ± 2) °C and (50 ± 10) % relative humidity before testing.

7.2 Test temperature

7.2.1 Unless otherwise agreed between the interested parties (for example for polyethylene), the temperature during insertion of the pin shall be (23 ± 2) °C.

7.2.2 The temperature during immersion shall be (23 ± 2) °C, unless otherwise specified or agreed upon by the interested parties, in which case the temperature can be (40 ± 1) °C or another temperature. Prior to immersion, the test specimens shall be stored in air at (23 ± 2) °C and (50 ± 10) % relative humidity. During storage in the reference environment (normally air), the same temperature shall be used as during immersion.

7.2.3 Tensile (preferred) or flexural testing shall be performed at (23 ± 2) °C and (50 ± 10) % relative humidity, or (23 ± 2) °C if the relative humidity is not critical.

7.3 Chemical medium

The chemical medium used for the test shall be that specified in the relevant International Standard. If there is no such specification, use either the chemical medium with which the material will be in contact in the expected application or a product agreed upon between the interested parties.

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8 Procedure

8.1 Cleanness

Test specimens, balls and pins shall be clean and free of grease, fat, perspiration and other substances that could affect the test result.

NOTE Exposure of test specimens to intense artificial light or sunlight could also affect the result.

8.2 Drilling the test specimens

8.2.1 General

Care shall be taken to avoid heating of the specimens during drilling. Select a drill speed, drill feed and drill geometry so that drilling produces a continuous chip that does not melt. Cool using an appropriate coolant (for example, oil-free compressed air, water or another coolant known to have no effect on the material under test). Specimens may be stored at temperatures lower than ambient before drilling. With ductile materials, this helps in controlling the dimensions of the hole.

8.2.2 Type A test specimen

Drill holes of diameter 2,8 mm in each test specimen and ream them to 3,0 mm. The holes shall be perpendicular to the surface of the test specimens, at least 15 mm apart and at least 15 mm from the edges of the test specimen.

NOTE Specimen preparation is difficult and critical and care is necessary.

8.2.3 Type B test specimen

Drill a hole of diameter 2,8 mm in each test specimen and ream the hole to 3,0 mm. Drill the hole perpendicularly to the surface of the test specimen, so that it passes through the intersection of the axes of symmetry to within 0,2 mm longitudinally and 0,02 mm transversely. Drill the set of test specimens for each deformation series consecutively with the minimum time delay.

NOTE To centre the hole when drilling, the type of fixture shown in Figure 3 is recommended.

8.2.4 Measurement of hole diameter

Condition the drilled and reamed test specimens for at least 24 h in the atmosphere specified in 7.2.2.

Measure the diameter of five holes selected at random to within 0,005 mm. Check that the range of values is less than 0,01 mm and then calculate the arithmetic mean. This mean value shall be taken as the hole diameter for the series.

8.3 Insertion of balls or pins

8.3.1 Definition of the deformation steps

If the failure limit is not known from previous tests or from experience, it is recommended that preliminary tests be performed using one specimen per deformation step.

NOTE Experience gathered testing amorphous plastics shows that the largest decrease in the indicative property occurs at a circumferential strain caused by impression of the ball or pin about 1/10th of the strain at yield in a tensile test. For the majority of plastics, strain at yield is less than 10 %. Based on a hole diameter of 3 mm, severe ESC damage can be expected to become visible at oversizes not larger than 0,3 mm.

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8.3.2 Balls

Insert one ball into each hole using a ball impression apparatus or other suitable means, for example the spindle feed of the drilling machine (see 5.8). Ensure that the position of each ball is symmetrical to the thickness of the test specimen to within ± 0,2 mm.

8.3.3 Pins

Insert the tapered end into the hole in the test specimen and press it in until there is firm contact between the pin and the wall of the hole along the entire length of the wall (see Figure 2). If several test specimens are mounted on the same pin, the distance between the test specimens shall be ⩾ 20 mm.

Do not wet the pin with the chemical medium selected for the test, since this will make it impossible to achieve reproducible exposure of the surface of the hole.

8.4 Immersion in the chemical medium

8.4.1 Conditioning

Store the prepared test specimen for (60 ± 5) min in the atmosphere specified in 7.2.2 prior to immersion in the test medium.

8.4.2 Short-duration test

8.4.2.1 Non-viscous liquid medium

Immerse the test specimens in the medium contained in a vessel (5.9) for up to 24 h at the specified temperature (see 7.2.2). Remove the specimens, wipe off the liquid using blotting-paper and allow the specimens to stand for (30 ± 5) min in the atmosphere specified in 7.2.2 before determining the stress-cracking behaviour.

8.4.2.2 Viscous medium

If the medium is viscous (for example paste or grease), cover the area of the hole on both sides of the test specimen with the medium. Store at the specified temperature up to 24 h, then wipe off the medium using blotting-paper and allow the specimens to stand for (30 ± 5) min in the atmosphere specified in 7.2.2 before determining the stress-cracking behaviour.

8.4.3 Long-duration test

Proceed as described in 8.4.2, exposing the test specimens to the medium for a specified or agreed period. If no period of contact is specified or agreed, it has been found convenient to carry out the test using periods of 1 day, 2 days, 4 days, 8 days, 16 days, etc., to determine the influence of time on the failure limit.

Before determining the failure limit (see 3.5), wipe off the medium using blotting-paper and allow the test specimens to stand for 2 h to 3 h in the atmosphere specified in 7.2.2.

NOTE 1 The immersion time of up to 24 h specified in 8.4.2.1 and 8.4.2.2 may be reduced if the medium is so aggressive that catastrophic failure occurs during the immersion period, and the failure limit cannot be determined in subsequent tests. For many chemical media, immersion times of 1 h have been found to be suitable.

NOTE 2 To obtain comparable results, especially in short-duration tests, it may be necessary to schedule impression of the balls or pins, immersion and testing in such a manner that the time between impression of the balls or pins and the test is equal for all oversize series in the test.

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8.5 Exposure in air

If a simultaneous test in air is to be performed, store the test specimens in the atmosphere specified in 7.2.2 for (24,0 ± 0,5) h for the short-duration test or for the test period(s) used for the long-duration test.

8.6 Determination of stress cracking

8.6.1 Type A test specimen

Determine the failure limit for products exposed to air and immersed in the chemical medium by visual observation, or by means of a lens of magnification ×5.

8.6.2 Type B test specimen

Determine the failure limit by the selected method (see 3.5).

The pin shall always be removed before flexural tests. For tensile tests, balls and pins may remain in the test specimens. When removing the balls or pins before the test, ensure that the removal process does not influence the test result.

NOTE Often the results of the flexural or tensile test on test specimens at deformation step zero are equivalent, whether determined in air or in a chemical medium. If the value determined in the chemical medium is higher, additional embrittlement should be suspected and, if it is lower, a softening of the material has occurred.

9 Expression of results

9.1 Type A test specimen

Record the oversize at which the first crack is visible. Record the time for the appearance of cracking either as the time when the first crack is detected or when half of the test specimens show at least one crack.

NOTE For routine testing, it is usually suffient simply to record whether or not a crack is visible after exposure under the chosen conditions, i.e. oversize, chemical medium and time.

9.2 Type B test specimen — Graphical evaluation

Plot the arithmetical means of the tensile or flexural values determined at each deformation step as ordinates against the oversize of the corresponding pin as abscissa. Draw a smooth curve through the points (see Figure 4 for an example).

Calculate the failure limit as follows:

Draw a line parallel to the X-axis through the point on the Y-axis corresponding to the failure limit. At the point of intersection of this line with the curve, draw a line perpendicular to the X-axis. The oversize value at the point of intersection of this line with the X-axis is the failure limit, in millimetres. Record this value to the nearest hundredth of a millimetre.

NOTE This may be done electronically using a suitable programme.

10 Precision

The precision of these methods is not known because interlaboratory data are not available in view of the variety of plastics materials and conditions. These methods may not be suitable for use in the event of disputed results as long as no precision data are available.

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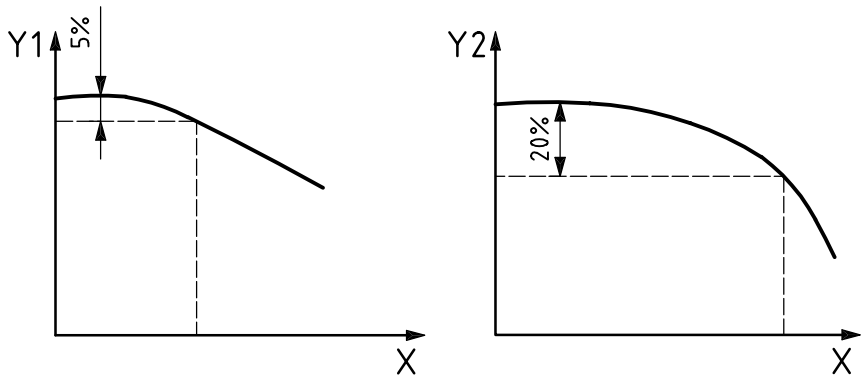

Key

X pin oversize, mm

Y1 tensile or flexural force at rupture

Y2 tensile elongation at rupture

Figure 4 — Example of graphical evaluation of results

11 Test report

The test report shall include the following particulars:

a) a reference to this part of ISO 22088;

b) all details necessary to identify the material tested;

c) the chemical medium used;

d) the test temperature;

e) the number of specimens tested (if applicable, in each direction of anisotropy) and their width and thickness;

f) the procedure used for preparation of the specimens and, whenever relevant, the elapsed time between their preparation and the beginning of testing;

g) the state of the specimens;

h) a description of the conditioning method used, and the conditioning atmosphere and time.

In addition, the following shall be included, depending on the test method used:

a) details of the deformation series [the mean diameter of the holes (see 8.2.4) and the diameters of the oversize balls or pins (5.6) used];

b) for test specimen type A only:

   1) the oversize at which the first crack was observed, and the time to the appearance of cracking,

   2) for routine testing:

      - the test conditions, i.e. oversize, medium and time,

      - whether cracks were visible or not;

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c) for test specimen type B only:

   1) the mechanical test used,

   2) the failure criterion used and the oversize value recorded as the failure limit in 9.2 for each method used, for example: ESC, failure criterion B2 (23 °C, 2 days), 3,35 mm,

   3) a graphical presentation of the results as described in 9.2,

   4) the relative stress-cracking factor, if requested, together with the reference environment used;

d) the date of testing.

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ICS 83.080.01

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