INTERNATIONAL STANDARD

ISO 13274

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Packaging — Transport packaging for dangerous goods — Plastics compatibility testing for packaging and IBCs

Emballages — Emballages de transport pour marchandises dangereuses — Essais de compatibilité des matières plastiques pour emballages et GRVs

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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 13274 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 261, *Packaging*, in collaboration with Technical Committee ISO/TC 122, *Packaging*, Subcommittee SC 3, *Performance requirements and tests for means of packaging, packages and unit loads*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This first edition of ISO 13274 cancels and replaces ISO 16101:2004[1)] and ISO 23667:2007[2)], which have been technically revised.

1) ISO 16101:2004, *Packaging — Transport packaging for dangerous goods — Plastics compatibility testing*.

2) ISO 23667:2007, *Packaging — Transport packaging for dangerous goods — Rigid plastics and plastics composite IBCs — Compatibility testing*.

Introduction

This International Standard was developed to provide requirements and test procedures to meet the compatibility provisions for plastics packagings and Intermediate Bulk Containers (IBCs) to contain liquids as set out in:

— The European Agreement concerning the International Carriage of Dangerous Goods by Road (ADR) (covering most of Europe) [1] and

— Regulations concerning the International Carriage of Dangerous Goods by Rail (RID) (covering most of Europe, parts of North Africa and the Middle East) [2].

This procedure is an alternative option to that set out in the UN Recommendations on the Transport of Dangerous Goods.

Plastics packaging/IBC material can be attacked by the chemical contents of the package. Such effects are caused by different mechanisms such as environmental stress cracking (ESC) chemical degradation and/or swelling.

The UN Recommendations and the associated modal regulations require that all packagings/IBCs be assessed for compatibility with the substances which they are to contain. The UN text makes special reference to plastics packagings/IBCs for liquids. The procedure therein contains details of testing for six months at ambient temperature with the liquid to be carried. RID/ADR permits as an alternative the use of standard liquids to which this International Standard refers.

The UN Recommendations are given legal entity not only to ADR and RID but also to:

— The International Civil Aviation Organisations Technical Instructions for the Safe Transport of Dangerous Goods by Air (ICAO Tis) (worldwide) [3] and

— The International Maritime Dangerous Goods Code (IMDG Code) (worldwide) [4].

The application of this International Standard will need to take account of the requirements of these international agreements and the relevant national regulations for domestic transport of dangerous goods as required by Directive 2008/68/EC of the European Parliament and council, as modified by Commission Directive 2012/45/EU of 3 December 2012 [5].

Although not stipulated in the UN Recommendations or the model regulations, these tests may be applied, where deemed appropriate, to polyethylene inner packaging of combination packaging.

Packaging — Transport packaging for dangerous goods — Plastics compatibility testing for packaging and IBCs

WARNING — The use of this International Standard could involve hazardous materials and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies the requirements and test methods for compatibility testing of plastics packagings/Intermediate Bulk Containers (IBCs) and composite packagings/IBCs with plastics inners containing liquids. The testing involves storage with the liquid to be transported. For polyethylene-based packaging, testing with a standard liquid as defined in *The European Agreement concerning the International Carriage of Dangerous Goods by Road* may be undertaken. Annex B describes small-scale laboratory tests that may be used to determine the assimilation of those products to be carried with the standard liquids.

Where there is any contradiction between this International Standard and any applicable regulation, the regulation always takes precendence.

2 Normative references

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

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

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

ISO 1133-1, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method*

ISO 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pyknometer method and titration method*

ISO 1628-3, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 3: Polyethylenes and polypropylenes*

ISO 1872-2:2007, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

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

ISO 11403-3, *Plastics — Acquisition and presentation of comparable multipoint data — Part 3: Environmental influences on properties*

ISO 11542-2:1998, *Plastics — Ultra-high-molecular-weight polyethylene (PE-UHMW) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 16495:2013, *Packaging — Transport packaging for dangerous goods — Test methods*

ISO 16770, *Plastics — Determination of environmental stress cracking (ESC) of polyethylene — Full-notch creep test (FNCT)*

UNITED NATIONS. The United Nations Recommendations on the transport of dangerous goods — Model Regulations. ST/SG/A.C. 10/1/Rev.17: United Nations

3 Terms and definitions

For the purposes of this document, the terms and definitions given in the UN Recommendations, ST/SG/A.C.10/1Rev17 and the following apply .

3.1
plastics packaging
drum and jerrican made from plastics material and composite packaging with inner plastics receptacles

3.2
plastics IBC
rigid plastics intermediate bulk container and composite intermediate bulk container with inner plastics receptacle

3.3
packaged substance
<chemical product> dangerous liquid to be transported in the packaging/IBC

Note 1 to entry: Packagings/IBCs used for solid packaged substances, which can become liquid at temperatures encountered during transport, also meet the requirements of packagings/IBCs for liquids.

3.4
standard liquid
defined liquid that is representative in its effect of a specific kind of interaction between a packaged substance and the plastics packagings/IBCs

4 Test requirements

4.1 General

Plastics packagings/IBCs selected in accordance with Clause 5 shall be conditioned with the packaged substance or a standard liquid, to which the packaged substance is assimilated.

NOTE Standard liquids and their assimilations can be found in ADR [1] and RID [2].

For packaged substances which are not assimilated to a standard liquid, small scale laboratory tests (see Annex B) may be used to compare their impacts to those of standard liquids.

The packaged substance shall be less aggressive than the standard liquid to which it will be assimilated. In the event the effect is more aggressive than that of the standard liquids, the six month procedure shall be followed as given in 8.2, or alternatively, and with the exception of nitric acid > 55 %, the accelerated procedure, in accordance with 8.3.Where the six-month procedure is carried out the specifications of the packaged substance are recorded.

When the standard liquid is water, proof of chemical compatibility is not required.

4.2 Conditioning

Plastics packagings/IBCs shall be conditioned in accordance with Clause 8.

4.3 Post-conditioning inspection

At the end of the conditioning period the packagings/IBCs shall be inspected for leakage. Where no leakage is apparent testing in accordance with 8.4 shall be performed.

4.4 Drop test

The drop test shall be performed in accordance with Annex F of ISO 16495:2013.

4.5 Stacking test

The stacking test shall be performed in accordance with Annex I of ISO 16495:2013.

4.6 Hydraulic pressure test

The hydraulic pressure test shall be performed in accordance with Annex H of ISO 16495:2013.

4.7 Leakproofness test

The leakproofness text shall be performed in accordance with Annex G of ISO 16495:2013.

4.8 Bottom lift test

The bottom lift test shall be performed in accordance with Annex K of ISO 16495:2013.

4.9 Top lift test

The top lift test shall be performed in accordance with Annex L of ISO 16495:2013.

4.10 Vibration test

The vibration test shall be performed in accordance with Annex Q of ISO 16495:2013.

4.11 Permeability testing

With the exception of composite packagings having a plastics receptacle with outer steel drum, plastics packaging shall be tested for permeability. A suitable test method can be found in ADR and RID. This test is only required when the packagings described above are intended to transport benzene, toluene, xylene or mixtures and preparations containing these substances.

NOTE Some substances can lead to permeation of the product through the (plastics) wall of the packaging. In some cases these substances give such a high degree of swelling that the required performance of the packaging is not met. In other cases the performance criteria are met, but the high degree of permeation could lead, besides the loss of product, to a dangerous situation because of vapours with dangerous explosive or toxic concentrations. Barrier materials or surface modifications can be used to decrease the amount of permeation and thus the risk of a dangerous situation.

4.12 Equivalent testing

The test methods described in this International Standard shall be considered to be the reference test methods.

Alternative methods may be used to demonstrate compliance with relevant regulations provided that:

— their equivalency to the reference method can be demonstrated;

— their use is recorded in the test report;

— prior approval is obtained from the competent authority.

5 Selection and preparation of packagings/IBCs

Packagings and IBC's shall be selected and prepared for testing as specified in ISO 16495:2013, Clause 5

Concerning closure applications all tests for a particular liquid shall be carried out at the same torque.

NOTE 1 The closure torque can vary for different seals.

NOTE 2 If application of the specified closure torque affects the subsequent performance of the seal during the packaging testing then the specified closure torque can be applied after the conditioning period.

NOTE 3 When mechanical tests have been successfully performed, the stacking test can be waived for standard liquids on composite packagings with plastic inner receptacles and non-plastics outer packaging.

NOTE 4 When mechanical tests, in accordance with ISO 16495:2013 have been successfully carried out, it is not necessary to carry out bottom lift test, top lift test and vibration test, for all IBC types. The stacking is also not necessary for composite IBCs having a non-plastics outer structure that supports the stacking load.

6 Additional information to be provided for assimilation

The packagings/IBCs user (with the assistance, where appropriate, of the packagings/IBCs manufacturer and the test laboratory) shall identify the packaged substance. The assimilation process shall consist of identifying the plastics material concerned and its possible interactions, such as swelling, environmental stress cracking (ESC) and molecular degradation.

The specification forms for plastics packagings/IBCs should identify the material by polymer type and grade.

NOTE Where tests are carried out using the packaged substance, the test report can be applicable for other substances having equivalent or lesser chemical effects.

7 Facilities for testing

See ISO 16495:2013, Clause 7

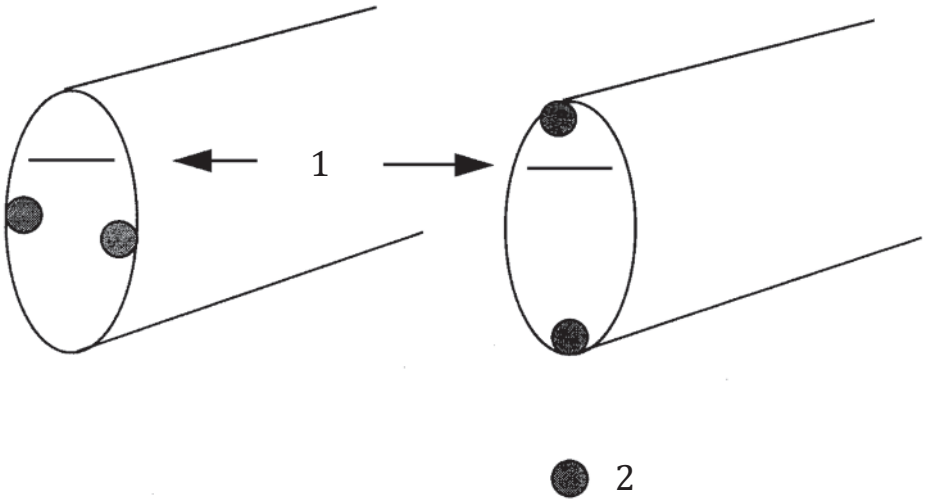
8 Conditioning procedures

8.1 General

After filling, the packagings shall be inverted for 24 h or 5 min if fitted with a vented closure and then restored to the normal standing position. At the end of the conditioning period as defined in 8.2 and 8.3, this inversion process shall be repeated.

The inversion process is not applicable for IBCs.

As an alternative to complete inversion the packaging may be laid on its side such that all closures are below the level of the substance being tested in accordance with Figure 1.



Key

1 liquid level

2 closure

Figure 1 — Explanatory diagram of alternative inversion method

8.2 Ambient conditioning

This test shall be carried out at ambient temperature for a period of 6 months.

For the purposes of this International Standard, ambient temperature, which shall be monitored and recorded, is considered to be not less than 15 °C.

NOTE The competent authority might, however, allow an extended period of test for temperatures below 15 °C.

8.3 Accelerated conditioning

The packagings/IBCs for test shall be conditioned for 21 days at a minimum temperature of 40 °C with each standard liquid, or a packaged substance, as relevant.

8.4 Procedure following the conditioning period

Following the conditioning period, all packagings/IBCs, except those intended to withstand the stacking test, shall be emptied, rinsed, inspected for damage and prepared for test in accordance with the test procedures for plastics packagings/IBCs for liquids specified in ISO 16495:2013. Testing shall commence within 21 days of the end of the conditioning period. If emptied the packagings/IBCs shall be kept closed until testing commences.

Packagings/IBCs which have been conditioned with standard liquid, *N*-butyl acetate, shall be emptied and refilled with a mixture of 1 %–10 % aqueous wetting agent solution and 2 % of *N*-butyl acetate for the stacking test.

NOTE 1 Where the closure elements (for example heat or induction seals) would have to be destroyed to empty the packaging after conditioning; the packaging is to be emptied through an additional opening drilled into the package. Such an opening is to be made so that the results of the other tests (drop, hydraulic pressure and leakproofness tests) are not affected.

NOTE 2 For substances presenting an unacceptable safety risk at 40 °C, it might be necessary to replace the filling substance by another substance where at least the same chemical interaction has been demonstrated and the agreement of the competent authority has been obtained.

The same closures and gaskets used during the conditioning of the packagings/IBCs shall be used for all of the testings, i.e. gaskets and closures shall not be replaced.

8.5 Reuse of standard liquids

The standard liquids shall be checked periodically in accordance with Table 1 as their effectiveness can be reduced over a period of time.

Table 1 — Reuse of standard liquids

Standard liquid	Specification
Wetting solution	New solution for each test or check surface tension against specification.
Acetic acid	Concentration (99 ± 1) %
Normal butyl acetate	≥ 98 % [a)]
Mixture of hydrocarbons	(16–21) % aromatic content [a)]
Nitric acid	Concentration ≥ 55 %
[a)] It is recommended that the absorption of these standard liquids is periodically checked with a control specimen of polyethylene of defined type and grade, in accordance with B.4.1 The used standard liquid is no longer fit for purpose when the determined absorption deviates by more than 5 % from the original determined value.	

Tests to monitor the quality of the standard liquids shall be done by appropriate means at intervals according to the frequency of usage.

9 Test report

The test report shall be written in accordance with Clause 4 of ISO 16495:2013.

Annex A
(informative)

Applicability of standard liquids to polyethylene types

A.1 Introduction

The standard liquid system has been developed for the investigation of the compatibility of high molecular weight high density polyethylene, but it may also be applied to other types of polyethylene and to packagings/IBCs produced from the above polyethylene types where the surface or surfaces have been fluorinated.

NOTE When closures or closure elements are manufactured from materials other than those referred to in A.2, alternative suitable methods to investigate compatibility will have to be employed.

A.2 Polyethylene types

A.2.1 High molecular weight high density polyethylene (PE-HD-HMW)

The natural relative (non-pigmented) density at 23 °C after annealing at 100 °C for 1 h shall be ≥ 0,940 g/cm^3 in accordance with ISO 1183-1.

The melt flow rate at 190 °C per 21,6 kg load shall be ≤ 12 g per 10 min in accordance with ISO 1133-1.

A.2.2 Medium molecular weight high density polyethylene (PE-HD-MMW)

The natural relative (non-pigmented) density at 23 °C after annealing at 100 °C for 1 h shall be ≥ 0,940 g/cm^3 in accordance with ISO 1183-1.

The melt flow rate at 190 °C per 2,16 kg load shall be ≤ 0,5 g per 10 min and ≥ 0,1 g per 10 min in accordance with ISO 1133-1,

or;

the melt flow rate at 190 °C per 5 kg load shall be ≤ 3,0 g per 10 min and ≥ 0,5 g per 10 min in accordance with ISO 1133-1.

A.2.3 Cross-linked polyethylene (PE-X)

PE-X is polyethylene having a changed chemical structure in which the major proportion of polymer chains are chemically connected with each other to form a three-dimensional network.

A.2.4 Linear medium density polyethylene

The natural relative (non-pigmented) density at 23° after annealing at 100 °C for 1 h shall be ≥ 0,927 g/cm^3 and ≤ 0,937 g/cm^3 in accordance with ISO 1183-1.

The melt flow rate at 190 °C per 2,16 kg load shall be ≥ 5,0 g per 10 min and ≤ 10,0 g per 10 min in accordance with ISO 1133-1.

Annex B
(normative)

Small-scale laboratory tests to assess packaged substances against standard liquids

B.1 Introduction

The small-scale laboratory tests listed as follows shall be used to assess whether a packaged substance can be assimilated to a standard liquid for polyethylene

Three tests cover specific interactions between the packaged substance and the plastics material. These are:

— Method A: absorption (one procedure);

— Method B: environmental stress cracking (three procedures);

— Method C: molecular degradation (three procedures).

NOTE Where alternative procedures are described each procedure is to be regarded as equivalent.

B.2 Requirements

B.2.1 Resistance to absorption (swelling)

For Method A (B.4.1) the percentage weight increase when tested with the packaged substance to be transported shall be less than or equal to that figure obtained when tested with the applicable standard liquid.

B.2.2 Resistance to environmental stress cracking

For Procedure B1 (B.4.2.2), the results shall demonstrate that with the packaged substance there is a lesser or equal effect than with the standard liquid used as a control.

For Procedure B2 (B.4.2.3) and Procedure B3 (B.4.2.4) the results shall demonstrate that with the packaged substance there is a time interval to failure equal to or greater than with the standard liquid used as a control.

B.2.3 Resistance to molecular degradation

For Procedure C1 (B.4.3.3) the melt flow rate of the specimen of the material in contact with the packaged substance shall not exceed that of the same material in contact with 55 % nitric acid.

For Procedure C2 (B.4.3.4) the viscosity number of the sample of the material in contact with the packaged substance shall not be less than that of the same material in contact with 55 % nitric acid.

For Procedure C3 (B.4.3.5) the elongation at break of the sample in contact with the packaged substance shall not be less than that with the same material with 55 % nitric acid.

B.2.4 Test report

A test report shall be prepared. The report shall include a full description of the packaged substance under test and the plastics material.

B.3 Selection and preparation of test specimens

B.3.1 A representative complete packaging (at least 48 h old) shall be supplied to the testing laboratory. Test specimens shall be prepared from material cut from this packaging.

NOTE By agreement with the competent authority the tests can also be carried out on test specimens prepared from compression moulded or extruded sheet produced from a specific polymer grade and specified thickness.

B.3.2 Each test specimen shall have a means of identification.

B.3.3 Each test specimen shall be examined for damage which could invalidate the tests.

EXAMPLE Surface imperfections or contamination are examples of damage that could invalidate the tests.

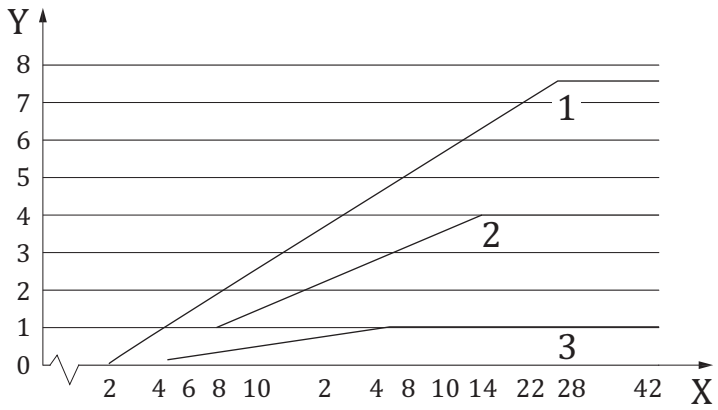
B.3.4 For cross-linked polyethylene, only test specimens taken from the packagings shall be used.

B.4 Test procedures

B.4.1 Resistance to absorption (Method A)

B.4.1.1 Principle

This method details the determination of the resistance to absorption of the plastics packaging when in contact with a packaged substance (see Figure B.1).



Key

Y increase in mass due to swelling, %

X storage period, *d*

1 mixture of hydrocarbons (white spirit)

2 normal butyl acetate

3 acetic acid

Figure B.1 — Determination of the absorption (increase in mass) of the samples immersed in the product at 40 °C

B.4.1.2 At least three test specimens of area not less than 450 mm^2 shall be cut from the centre of the container side wall or from a compression moulded or extruded sheet.

B.4.1.3 The initial mass of each of the test specimens (W_0) shall be recorded.

B.4.1.4 The test specimens shall be kept fully immersed in the packaged substance in a suitable receptacle.

B.4.1.5 Test specimens shall be immersed until absorption is complete, i.e. constant mass is reached. For normal test conditions with specimen thickness 2,0 mm or less and test temperature 40 °C, this is typically achieved within a test period of 28 days.

B.4.1.6 At the end of the test period or at appropriate test intervals remove the test pieces, remove all traces of surface liquid, and record the mass of each test piece (W_1).

B.4.1.7 Test specimens shall only be used once.

B.4.1.8 Results:

The mean of three results to two significant figures shall be recorded.

— % mass increase $\Delta W = \frac{100\,(W_1 - W_0)}{W_0}$

— W_0 = initial mass;

— W_1 = mass at end of test period.

B.4.1.9 Criteria for assessment

The percentage mass increase, when tested with the packaged substance, shall be less than or equal to that obtained when tested with the appropriate standard liquid.

NOTE This can be expected to be:

— up to 1 % for water, wetting agent solution, acetic acid, or nitric acid;

— approximately 4 % for *N*-butyl acetate;

— approximately 7,5 % for mixture of hydrocarbons (white spirit).

B.4.2 Resistance to environmental stress cracking (Method B)

B.4.2.1 General

One of the following three alternative procedures shall be used to determine environmental stress cracking:

i) pin impression test (see B.4.2.2)

ii) bent strip test (see B.4.2.3)

iii) full notch creep test (see B.4.2.4)

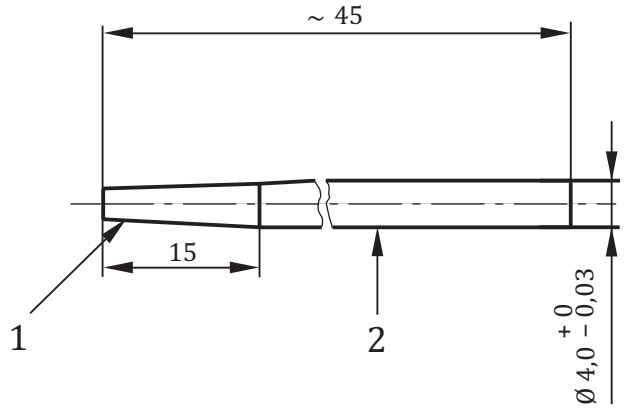
B.4.2.2 Pin impression test (Procedure B1)

B.4.2.2.1 Special equipment required for test

i) Polished pins, made from material resistant to the product under test, (e.g. stainless steel, glass) as specified in Figure B.2.

ii) Tool, for notching specimen to the required dimensions, in accordance with Figure B 3. The notch radius shall be ≤ 0,05 mm.

Dimensions in millimetres



Key

1 gradient approximately 1:10

2 polished

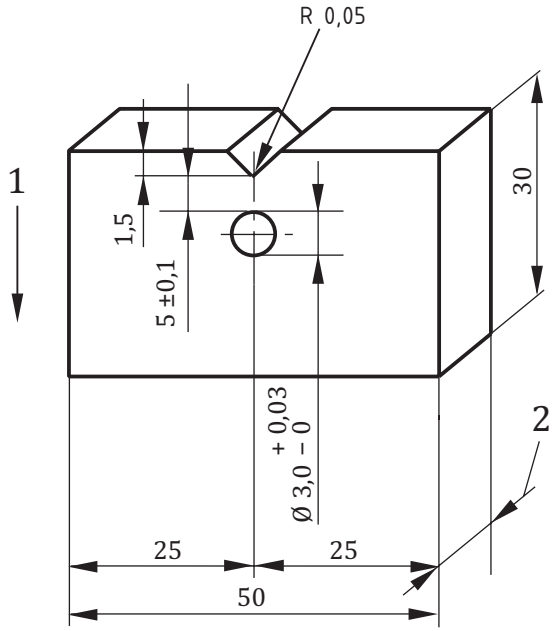
Figure B.2 — Polished pins for Pin Impression Test

B.4.2.2.2 Preparation of test specimens

Cut at least 70 specimens from a packaging, a compression moulded or extruded sheet. Each test specimen shall not be less than 50 mm long, 30 mm wide and 2 mm or greater in thickness. The test specimen shall be notched and a hole drilled (3 mm in diameter) in accordance with Figure B.3.

The distance between the bottom of the notch and the edge of the hole shall be (5 ± 0,1) mm or (4 ± 0,1) mm, the latter being used to shorten the testing time with certain grades of polyethylene.

Dimensions in millimetres



Key

1 direction of extrusion

2 5 mm dimension may be 4 mm where appropriate, in which case the 1,5 mm dimension is increased to 2,5 mm

Figure B.3 — Test specimen for Pin Impression Test

B.4.2.2.3 Preconditioning procedure

Test specimens shall be immersed in both the packaged substance and the standard liquid for a period of 21 days at either (40 ± 1) °C or a higher specified temperature controlled to ± 1 °C, the latter being used to shorten the test time with certain grades of polyethylene.

NOTE Where it has been shown that this pre-conditioning effect has no effect on test liquids and assimilated products, this step can be eliminated.

B.4.2.2.4 Stress cracking test

An equal number of specimens shall be immersed in both the packaged substance and the standard liquid; normally this is wetting solution or acetic acid.

NOTE *N*-butyl acetate can be used where it is intended to show the combined effect of stress cracking and absorption. This depends on the results of Method A (see B 4.1).

At the end of the storage period remove the test pieces, and put aside 10 specimens. Insert the polished pin (B.4.2.2.1) into the 3 mm hole in each of the remaining specimens. The pin shall penetrate the test piece until the parallel section of the pin is inserted into the hole.

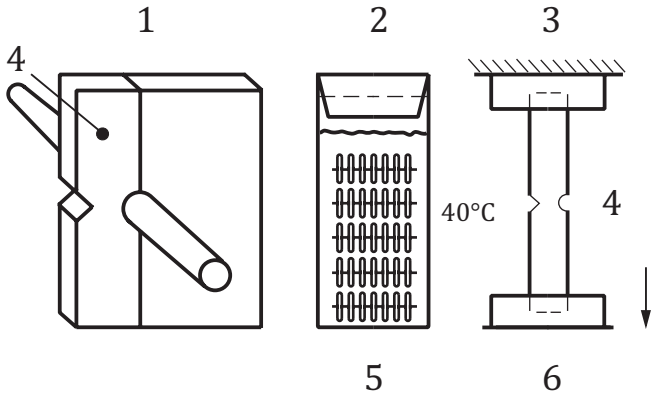
Return these pinned pieces to the liquids and immerse in accordance with B.4.2.2.3, except for *N*-butyl acetate where the stress cracking test shall be performed in a mixture of (1-10) % aqueous wetting agent solution mixed with 2 % *N*-butyl acetate (see B.2.3).

At appropriate intervals remove 10 pieces and allow to cool to room temperature. Remove the pins carefully. Cut each across the 3 mm hole parallel to the notched edge in accordance with Figure B.4.

Perform a tensile test on the notched part of each of the test pieces no longer than 8 h after removal from the test liquid.

Determine the tensile strength in accordance with ISO 527-2 at (23 ± 2) °C with a testing speed of 20 mm/min.

Calculate the mean tensile strength of each set of test pieces. Graphically plot the residual tensile strength as a percentage of the tensile strength of the original 10 test pieces which were put aside after pre-storage.



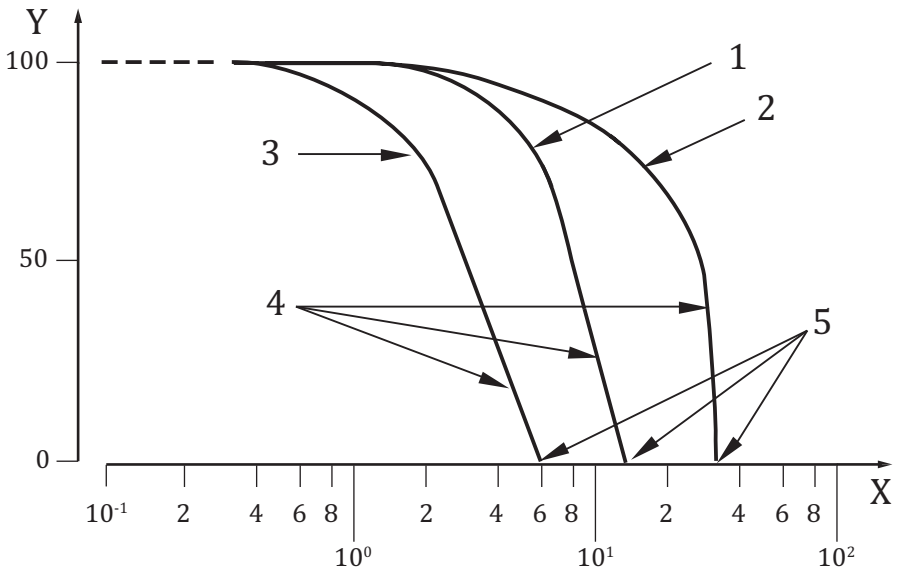
Key

1 test sample with pin
2 storage in filling substance
3 tensile test
4 test sample section A
5 40° C or 50° C where appropriate
6 testing speed v = 20 mm/min

Figure B.4 — Pin Impression Test: specimen preparation, storage and testing

B.4.2.2.5 Criteria for assessment

Compare the curves to determine whether the packaged substance has a stronger or weaker effect than the standard liquid, in accordance with Figure B.5.



Key

Y residual tensile strength σ_{max} %

X storage period, *d*

1 standard liquid

2 filling substance 2 (less aggressive than standard liquid)

3 filling substance 1 (more aggressive than standard liquid)

4 residual tensile strength curves

5 time standing until test sample is cracked through

Figure B.5 — Pin Impression Test

An alternative visual method of assessment of specimen failure times may be used, as follows:

15 samples prepared in accordance with B.4.2.2.2 are preconditioned in accordance with B.4.2.2.3 and subjected to the stress cracking method in accordance with B.4.2.2.4. By visual checks the time for cracking is determined for each pinned test sample (the crack usually propagates from the tip of the notch to the pin). The criterion for assessment is based on the time for 8 of the 15 specimens in the standard liquid to have failed (*T*). For the packaged substance, this time shall not be less than *T*.

This alternative method may be used with the approval of the competent authority.

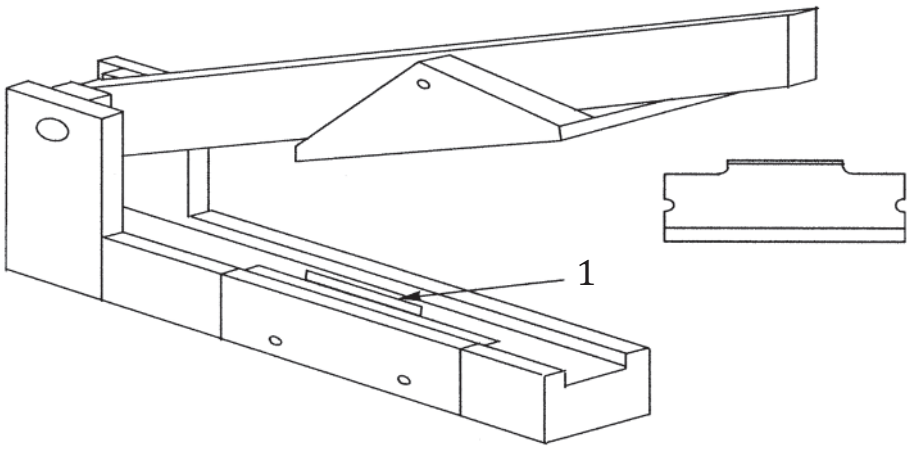
B.4.2.3 Bent strip test — 'Bell Telephone Test' (Procedure B2)

B.4.2.3.1 Principle

This procedure uses specimens with a controlled imperfection (notch) in accordance with ASTM D 1693-00 [6].

B.4.2.3.2 Special equipment

Slot notching tool, transfer tool, bending tool, in accordance with Figures B.6, B.7 and B.8.



Key
1 blade

Figure B.6 — Slot notching tool

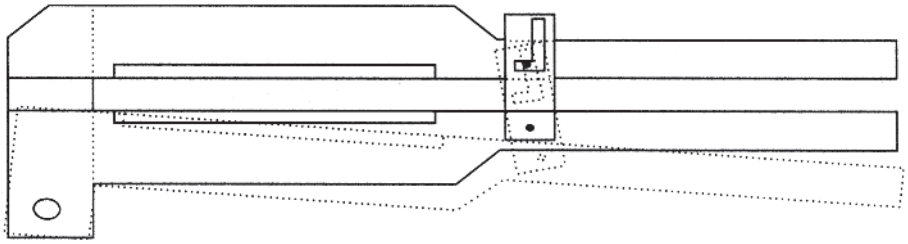
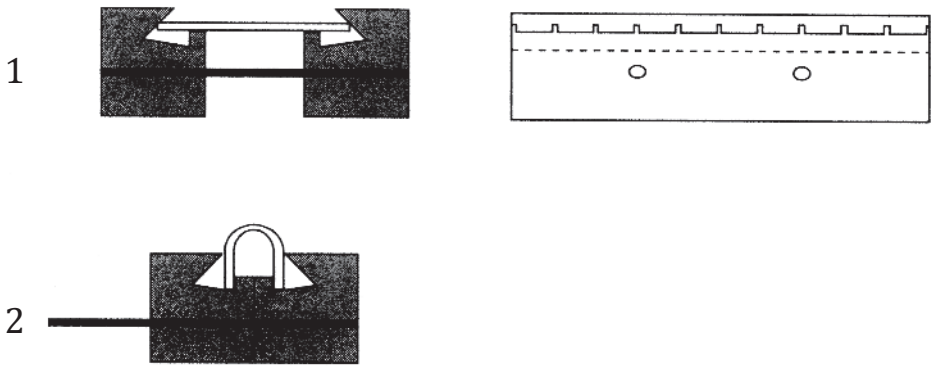


Figure B.7 — Transfer tool



Key
1 clamp open with flat specimen
2 clamp closed with bent specimen

Figure B.8 — Specimen bending tool

B.4.2.3.3 Preparation of test specimens

A suitable number of test specimens shall be cut from the packaging side wall, or from a compression moulded or extruded sheet. Test specimens cut from the container shall be subsequently compression moulded, using a specified moulding temperature and cooling rate in accordance with ISO 1872-2:2007, method B.

The finished thickness shall be 1,875 mm ± 0,125 mm.

Cut out 10 test specimens of dimensions (38,0 ± 2,5) mm × (13,0 ± 0,8) mm from the moulded sheet with a sharp cutting die. Do not cut test specimens from within 10 mm of the edge of the moulded sheet. Check that all the test pieces are within the specified thickness tolerances.

Using a sharp blade and jig, notch each test specimen centrally over a length of 19,05 mm ± 0,15 mm and a depth of 0,35 mm ± 0,05 mm.

B.4.2.3.4 Pre-conditioning procedure

Immerse the test specimens in the product under evaluation for 21 days at 40 °C or a higher specified temperature controlled to ± 1,0 °C.

NOTE Where it has been shown that this pre-conditioning has no effect on test liquids and assimilated products, this step can be eliminated.

B.4.2.3.5 Stress cracking test

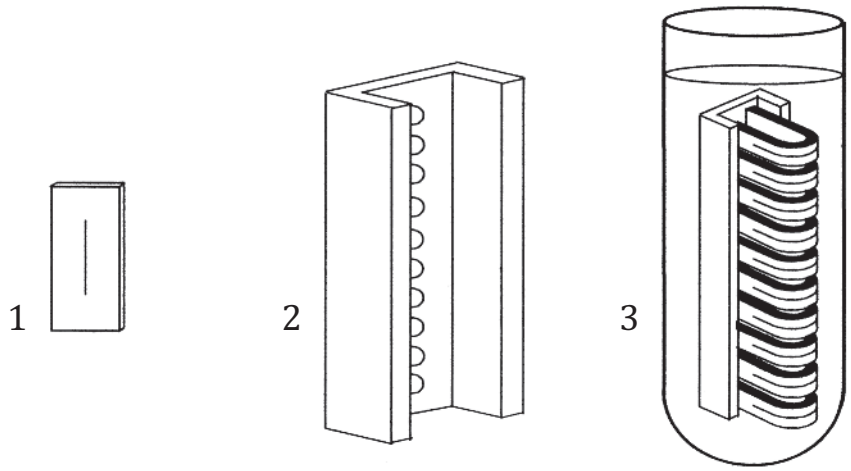
Place the test specimens, notch side upwards in a bending clamp and, using a vice, close the clamp over a period of approximately 30 s.

Using a transfer tool, carefully remove the test specimens from the clamp and place them in the specimen holder, a channel of length 165 mm, depth 10 mm, and internal width (11,75 ± 0,05) mm. Gently press down the test specimens to ensure that they are all firmly against the base of the channel in accordance with Figure B.9.

Place the channel in a suitable receptacle and cover it with the product under evaluation. Store at a temperature of 40 °C or a higher specified temperature controlled to ± 1 °C.

Inspect each test piece at suitable test intervals (normally daily) and record as a failure any test piece that shows a visible defect, usually a crack running at right angles to the notch.

The test shall be terminated when all specimens have failed or after 1 000 h.



Key

1 test specimen
2 specimen holder
3 specimens in test assembly

Figure B.9 — Bent Strip Test: test specimen, holder and assembly

B.4.2.3.6 Criteria of acceptance

The time for 50 % of specimens to fail (ƒ50 value) in the packaged substance shall be ≥ ƒ50 in the standard liquid.

B.4.2.4 Full notch creep test (FNCT) (Procedure B3)

B.4.2.4.1 Principle

A test specimen in the form of square section bar with coplanar notches in each face at the centre is subjected to a static tensile load in a temperature controlled environment in accordance with ISO 16770. The geometry of the test specimen is such that plane conditions are obtained and brittle failure occurs under appropriate load and temperature conditions. The time for this brittle failure to occur after loading is recorded.

B.4.2.4.2 Terms and definitions

B.4.2.4.2.1

failure

complete separation of the two halves of the test specimen

B.4.2.4.2.2

brittle failure

failure where the fracture surface exhibits no permanent material deformation to the naked eye

EXAMPLE Stretching, elongation and necking down; see Figure B.10.[1]

NOTE In tougher materials an extended ligament might form in the centre; see Figure B.10.[2]

B.4.2.4.2.3

ductile failure

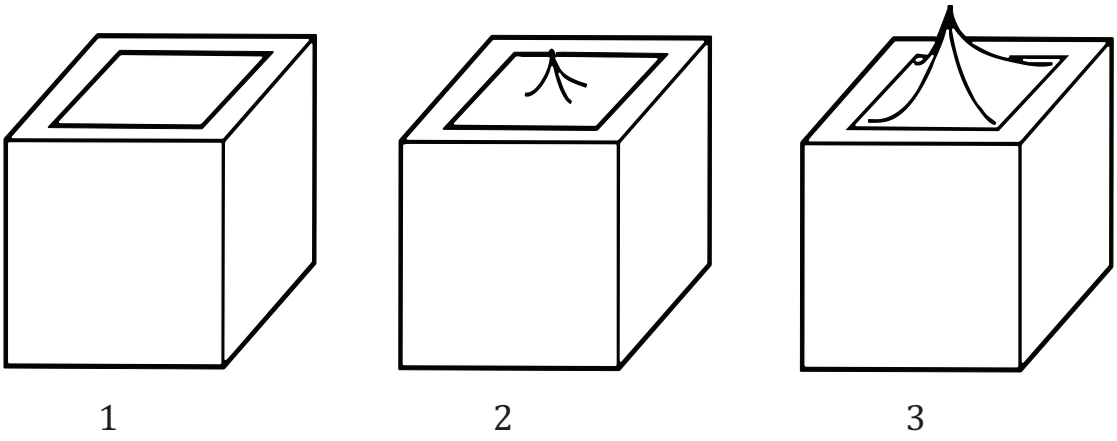
failure where the fracture surface clearly exhibits permanent material deformation with stretching, elongation and necking down

NOTE See Figure B.10.[3]

B.4.2.4.2.4

ligament area

remaining cross-sectional area after notching



Key

1 brittle
2 brittle
3 ductile

Figure B.10 — Fracture surfaces

B.4.2.4.3 Apparatus

B.4.2.4.3.1 Loading device

The load shall be applied by a device with a lever arm loading machine with an arm ratio between 4:1 and 10:1.

EXAMPLE A typical example of such a device is shown in Figure B.11.

The lever arm ratio *R* shall be equal to L1/L2. When the lever arm is fitted with the top specimen grip and the weight carrier it shall be horizontal, i.e. balanced.

The specimen grips shall be designed to prevent slippage of the test specimen and ensure that the load is transmitted axially through the test piece.

EXAMPLE This can be achieved via a low friction universal coupling to prevent bending of the test specimen during the test. A typical test specimen grip assembly is shown in Figure B.12.

In addition to the above example, the tensile load may be applied directly using dead weights or any other means for producing a constant load.

The loading device should be capable of applying the load to an accuracy of ± 1,0 %.

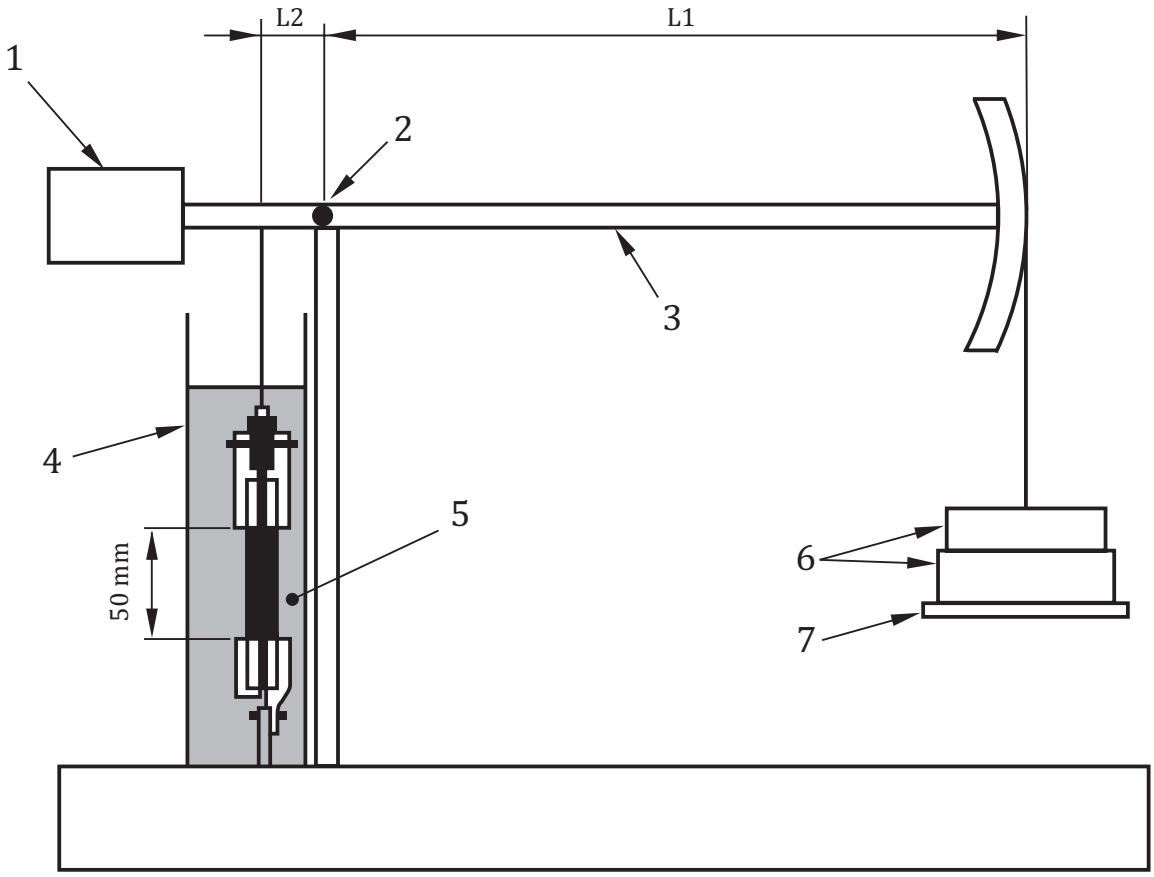
NOTE 1 The balanced loading apparatus as described in ISO 22088-2 has also been used satisfactorily.

The functioning and calibration of the equipment shall be checked on a regular basis because the applied load is a critical parameter.

NOTE 2 The calibration of a lever arm machine can be checked by hanging a series of known weights on the specimen side of the lever arm and counterbalancing these in turn with weights on the weight hanger. The ratio of the former to the latter provides a direct measure of the arm ratio and hence a check on the operation of the machine.

In the case of multiple specimen testing, care shall be taken to avoid undue disturbance of the remaining test specimens, when one or more of the other test specimens fail.

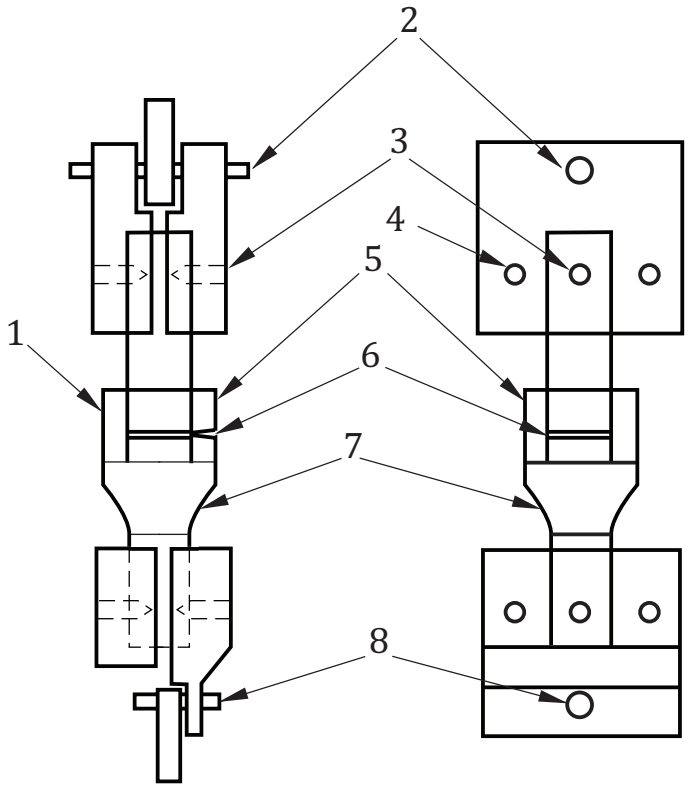
NOTE 3 Measurement of the extension of the test piece or movement of the lever arm provides useful information. The rate of extension of the test specimen will increase when the initiation of the crack from the notch has occurred and will increase rapidly when failure is imminent.



Key

1 counterweight
2 low friction roller or knife edge
3 balance lever arm
4 example of environmental chamber
5 environment
6 weights
7 weight hanger

Figure B.11 — Loading device



Key

1 small environmental chamber
2 coupling pin
3 grub screws to prevent slipping
4 clamp bolts
5 glass tube
6 notch
7 heat shrink tube
8 coupling pin

Figure B.12 — Specimen grip assembly

B.4.2.4.3.2 Thermostatically controlled environment

A suitable chamber shall be designed to contain the environment and ensure full immersion of the test specimen(s). The chamber shall be constructed of material(s) which do not affect the environment or vice versa. The temperature of the environment shall be controlled to maintain the test specimens within ± 1,0 °C of the specified test temperature. Where the environment is aggressive, the chamber can be very small as shown in Figure B.12 with the test specimen grip assembly.

NOTE When the environment is likely to separate, constant agitation is required.

B.4.2.4.3.3 Temperature measuring device

A calibrated thermometer, thermocouple or thermistor with an accuracy of ± 1,0 °C.

B.4.2.4.3.4 Timing device

This shall automatically stop or record the point when the test specimen fails by either fracture or excessive displacement of the grips. The accuracy of the timing equipment shall be ± 1 min.

B.4.2.4.3.5 Notching apparatus

The machine shall be designed so that the notches are coplanar and the plane of notching is perpendicular to the tensile axis of the test specimen. The machine shall have a device to ensure that the notches are placed in the centre of the test specimen. Razor blades shall be used provided their notch tip radius is less than 10 µm. A cutting machine with a tool, like a broaching device, is also acceptable as an alternative, provided the notch tip radius is also less than 10 µm.

NOTE A device, appropriately dimensioned, as illustrated in ISO 11542-2:1998, Figure B.1, would be satisfactory.

B.4.2.4.3.6 Microscope

A microscope is required to allow accurate measurement of the actual ligament dimensions (distance between the tips of the notches) after failure. It shall be read to an accuracy of ± 100 µm.

B.4.2.4.4 Preparation of test specimens

B.4.2.4.4.1 Test specimen geometry

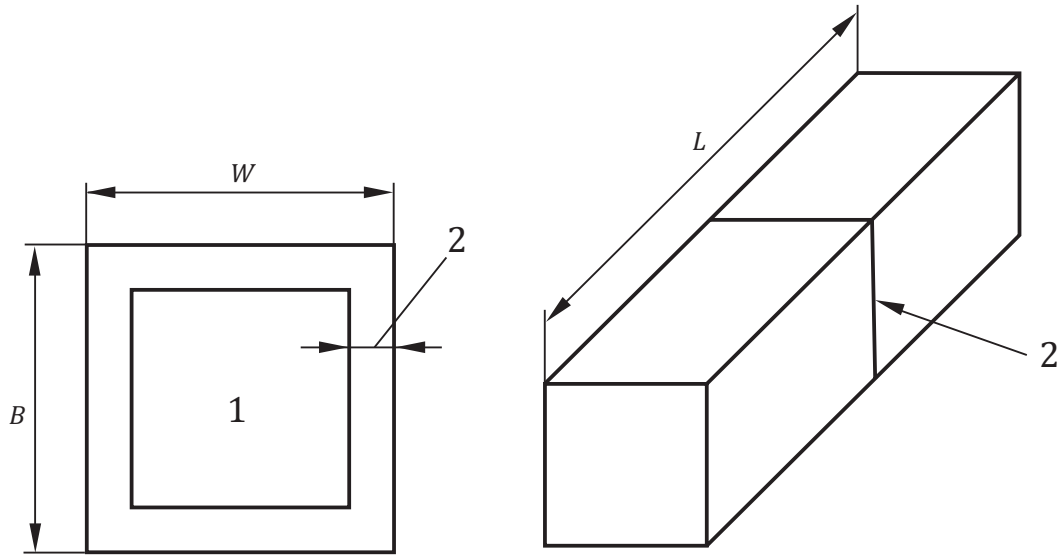
The test specimen geometries shall be in accordance with Table B.1. If other specimens are used, these shall be made so that the ligament area is approximately 50 % of the total cross-sectional area of the specimen in accordance with Figure B.13.

NOTE This is to make sure that specimen failure will occur under the specified conditions.

Table B.1 — Test specimen geometry

	Specimen dimensions (mm) **Length × Width × Breadth**	**Notch depth** (mm)	**Stress** (MPa)	**Temperature** (°C)
A	100 × 10 × 10	1,60	4,00 or 6,00	80
B[a]	90 × 6 × 6	1,00	9,00	50
C	90 × 6 × 6	1,00	12,00	23

[a] Test specimen B, 90 × 6 × 6 mm, with 1 mm notch depth is recommended.



Key

W width
L length
B breadth
1 ligament area
2 notch

Figure B.13 — Test specimen showing notch and ligament area

B.4.2.4.4.2 Test specimen preparation

Test specimens, for material testing, shall be prepared from compression moulded sheet. ISO 1872-2:2007 method B or ISO 11542-2:1998, Table 1 shall be used as appropriate. These standards specify moulding and cooling conditions. Machine the test specimens to size from the moulded sheet in accordance with ISO 2818. Trim the specimen edges of any remaining swarf left after machining. Test specimens cut from extruded or moulded finished goods shall be machined according to ISO 2818.

B.4.2.4.4.3 Test specimen notching

Specimens shall be notched at room temperature. Due care shall be taken to avoid blunting the notch during manufacture, e.g. use of excessive speed/force, as this will invalidate the results. If a razor blade is used it shall be used for notching no more than one hundred notches. Whichever device is used for notching, the tolerance on the required notch depth is ± 0,1 mm. Notch integrity shall be inspected microscopically.

B.4.2.4.4.4 Conditioning of test specimens

Notched specimens shall be stored at (23 ± 2) °C in accordance with ISO 291. When they are required for use at other temperatures they shall be conditioned in the environment at the test temperature for 1,0 h after clamping in the loading apparatus, prior to loading.

NOTE If other than the recommended test specimens (90 × 6,0 × 6,0) mm specimens are used, for thicker section specimens a longer conditioning period might be required.

B.4.2.4.5 Test procedure

B.4.2.4.5.1 Choice of stress and temperature

Select a stress and temperature from Table B.1 which will cause brittle failure of the test specimens. It is advisable to test a number of specimens, for example 4, with nominal stresses above and below the selected value; this is to compensate for variability in ligament area introduced during the notching operation. For example at a selected stress of 9 MPa, a series of nominal stress values such as 8,25, 8,75, 9,25 and 9,75 MPa could be used.

A stress of 9,0 MPa at 50 °C using Specimen B, (90 × 6,0 × 6,0) mm with 1,0 mm notch depth, is recommended.

B.4.2.4.5.2 Calculation of test load

The test load is calculated from the formula:

$$M = \frac{A_n \sigma}{9{,}81\, R}$$

where

M is the applied mass in kilograms;

A_n is the nominal ligament area in mm^2;

σ is the required tensile stress in MPa;

R is the lever arm ratio (Figure B.11), which equals one for a dead weight system.

B.4.2.4.5.3 Application of load to test specimen

The notched test specimen is placed in the grips of the lever loading machine (Figures B.11 and B.12), taking care to avoid bending and twisting the specimen. The test specimen shall be positioned with half its length free between the grips with the notch plane located in the centre. The whole specimen located in the grips shall be immersed in the environment and conditioned to the temperature specified in Table B.1. After conditioning, the calculated load is gradually applied to the lever arm avoiding shock loading of the test specimen. At the same time the clock or timing device shall be activated.

NOTE 1 It is convenient to lower the weight carrier using a suitable jack or other means.

NOTE 2 Lower temperatures will increase the time to failure of the test specimen. Higher temperatures will decrease the time to failure but if too high a temperature is used, changes in crystallinity and possible oxidative ageing might occur. The same will apply when different environments are used.

B.4.2.4.5.4 Calculation of results

The fracture surface of each test specimen is examined to ensure that it is of the brittle type (see Figure B.10). The dimensions of the ligament are measured using a travelling microscope and the ligament area is calculated.

The applied stress, σ_L, is given by the formula:

$$\sigma_L = \frac{9{,}81RM}{A_L}$$

where

σ_L is the corrected tensile stress in MPa;

A_L is the measured ligament area in mm^2;

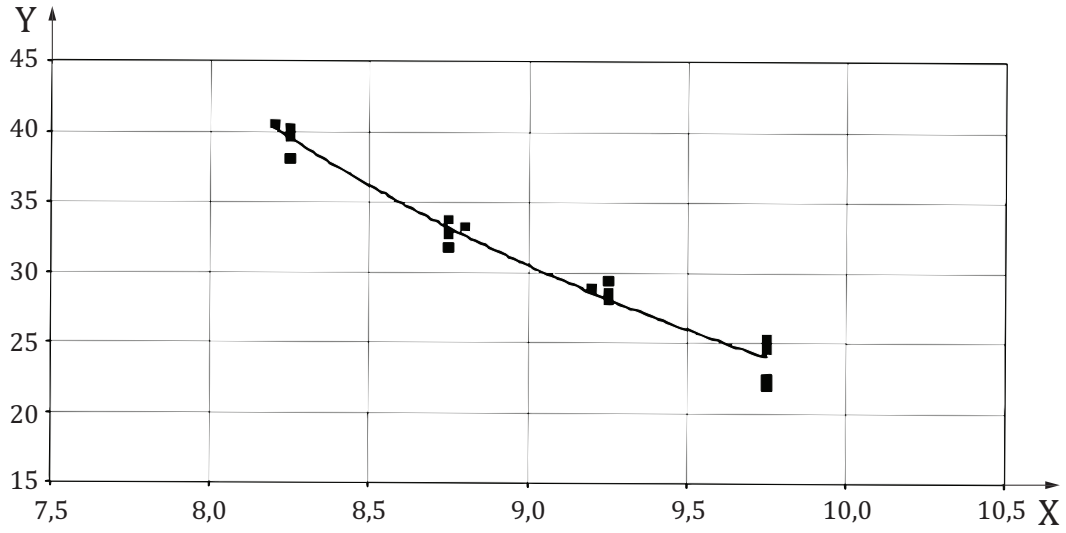
M is the applied mass in kilograms;

R is the lever arm ratio (Figure B.11) which equals one for a dead weight system.

The time to failure is plotted against the actual applied stress and the failure time at the reference stress interpolated from the graph.

B.4.2.4.6 Precision and reproducibility

At the present time there is no international agreement on precision and reproducibility. The precision of this test method is not known and an indication of repeatability is given in Figure B.14. It is hoped that a full precision statement will be given in a later revision of ISO 16770. For a reference stress of 9 MPa, the failure time is given as 30,5 h with 95 % confidence limits of ± 0,5 h. The standard deviation from the regression line is 1 h.



Key

X time (h)

Y applied stress (MPa)

Figure B.14 — Indication of repeatability

The major sources of error are:

a) load is applied too quickly, blunting of the notch may occur, rendering the results invalid;

b) notch is too blunt after notching;

c) notches are not co-planar;

d) tolerances on environment temperature are not met;

e) environment has aged or has not been stirred.

B.4.2.4.7 Criteria of acceptance

The time to failure in the packaged substance shall be greater than the time to failure in the standard liquid.

B.4.2.4.8 Test report

The test report shall include the following information:

a) Reference to this International Standard;

b) all details necessary for complete identification of the test material, e.g. manufacturer, production data, etc.;

c) all details necessary for the identification of the test specimen, e.g. cut from compression moulded sheet, or from a container;

d) the actual stress, based on the ligament area, used on the test specimen;

e) time to failure in comparison to the standard liquid or duration of test if failure has not occurred;

f) specimen dimensions as given in Table B.1;

g) environment temperature and concentration;

h) full details of the environment used;

i) any variations introduced not in the standard, e.g. notching procedure;

j) date and time for start and end of the test;

k) method of notching, i.e. razor blade or broach.

B.4.3 Resistance to molecular degradation (Method C)

B.4.3.1 General

One of the three alternative procedures shall be used for determining the resistance to molecular degradation:

— Procedure C1: measuring melt flow rate;

— Procedure C2: measuring viscosity number;

— Procedure C3: measuring elongation to break.

B.4.3.2 Applicability of method

(i) If the packaged substance causes absorption < 1 %, in accordance with the procedure in B.4.1, the specimen shall be dried (e.g. in a vacuum storage oven at 50 °C) until the mass remains constant to a level < 1 % before measuring the MFR.

(ii) If the packaged substance causes higher absorption ratios after drying, this implies that Procedure C1 is not applicable and in this case Procedure C2 or Procedure C3 shall be used.

(iii) If the packaged substance (e.g. organic peroxide) penetrates into the PE specimen and leads to cross-linking at elevated temperatures (MFR measurement conditions), Procedure C3 shall be used. Procedures C1 and C2 are not applicable.

(iv) For cross-linked PE (PE-X) Procedure C3 shall be used. Procedures C1 and C2 are not applicable.

B.4.3.3 Melt flow rate (Procedure C1)

B.4.3.3.1 Cut a suitable number of test specimens of an area not less than 450 mm^2 from the packaging side wall or from a compression moulded or extruded sheet. Identical specimen dimensions shall be used for the samples to be immersed in both the packaged substance under evaluation and the standard liquid 55 % nitric acid.

B.4.3.3.2 Determine the initial MFR of a minimum of three test specimens in accordance with ISO 1133-1.

B.4.3.3.3 For each evaluation, place a minimum of three test specimens per test interval as specified in B.4.3.2.4 in a suitable receptacle and immerse them in the product under evaluation. Store the receptacle at 40 °C or a higher specified temperature controlled to ± 1,0 °C.

B.4.3.3.4 After 21 days, and subsequently at 7-day intervals for a total storage period of up to 42 days, remove a set of at least three pieces. Carefully wash and dry the specimens and condition them in a vacuum oven under conditions sufficient to remove residual product, until the mass of the specimen remains constant to a level < 1 %.

B.4.3.3.5 Measure the MFR of each set of test specimens in accordance with ISO 1133-1.

B.4.3.4 Viscosity number (VN) (Procedure C2)

B.4.3.4.1 Cut a suitable number of test specimens of an area not less than 450 mm^2 from the centre of a container side wall or from a compression moulded or extruded sheet. Identical specimen dimensions shall be used for the samples to be immersed in both the packaged substance under evaluation and the standard liquid 55 % nitric acid.

B.4.3.4.2 Determine the initial VN of a minimum of three test specimens in accordance with ISO 1628-3.

B.4.3.4.3 For each evaluation place a minimum of three test specimens per test interval in a suitable receptacle and immerse them in the product under evaluation. Store the receptacle at 40 °C or a higher specified temperature controlled to ±1,0 °C.

B.4.3.4.4 After 21 days, and subsequently at 7-day intervals for a total storage period of up to 42 days, remove a set of at least three specimens. Carefully wash and dry them and condition them in a vacuum oven under conditions sufficient to remove any residual products.

B.4.3.4.5 Measure the VN of each set of test specimens in accordance with ISO 1628-3.

B.4.3.5 Elongation to break (Procedure C3)

B.4.3.5.1 Cut a suitable number of test specimens to ISO 527-2 Type 5 or 1B, in accordance with ISO 11403-3 from the centre of a packaging side wall in the extrusion direction. Identical specimen dimensions shall be used for the samples to be immersed in both the packaged substance under evaluation and the standard liquid 55 % nitric acid.

B.4.3.5.2 In accordance with ISO 527-2 use a tensile testing machine to determine the initial percentage elongation to break of 5 specimens at an elongation rate of v = 100 mm/min.

B.4.3.5.3 For each evaluation, place a minimum of 5 test specimens per test interval in a suitable receptacle and immerse them in the product under evaluation. Store the receptacle at 40 °C or a higher specified temperature controlled to ± 1,0 °C.

B.4.3.5.4 After 21 days, and subsequently at 7-day intervals for a total storage period of up to 42 days, remove a set of at least 5 specimens. Carefully wash and dry them and allow them to reach equilibrium at (23 ± 1) °C prior to testing.

NOTE Other periods of testing can be applied where prior experience has shown them to be suitable, provided they are stated in the test report.

B.4.3.5.5 Measure the percentage elongation to break of each set of test specimens in accordance with ISO 527-2.

B.4.3.6 Criteria of assessment

The packaged substance shall be assimilated to the standard liquid 55 % nitric acid if it causes equal or less damage, i.e. the specimens show in any of the following tests:

— Procedure C1: equal or lower increase in melt flow rate (MFR);

— Procedure C2: equal or lower decrease in viscosity number (VN);

— Procedure C3: equal or less reduction in elongation to break.

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