International Standard 4433

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Polyolefin pipes — Resistance to chemical fluids — Immersion test method — System for preliminary classification

Tubes en polyoléfines — Résistance aux fluides chimiques — Méthode d'essai par immersion — Système de classification préliminaire

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4433 was developed by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, and was circulated to the member bodies in May 1983.

It has been approved by the member bodies of the following countries :

Australia	India	Romania
Austria	Israel	South Africa, Rep. of
Belgium	Italy	Spain
Canada	Japan	Sweden
Czechoslovakia	Morocco	Switzerland
Finland	Netherlands	United Kingdom
France	Norway	USA
Germany, F. R.	Poland	USSR

No member body expressed disapproval of the document.

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Contents

Polyolefin pipes — Resistance to chemical fluids — Immersion test method — System for preliminary classification

0 Introduction

Because of their varied applications, polyolefin pipes are frequently required to convey or be in contact with chemical products, fuels, lubricants, etc. and sometimes their vapours.

Under the action of a liquid, the wall of a polyolefin pipe can be the location for several concurrent phenomena; on the one hand, absorption of liquid and/or extraction of its soluble constituents from the pipe walls into the liquid; on the other hand, a chemical reaction usually involving a significant change in the properties of the pipe. The phenomena also differ according to the external and internal stresses affecting the pipes conveying the products (temperature, pressure, wall thickness, etc.).

By stresses are meant those forces caused by internal or external factors such as temperature, variation of temperature, inside pressure, bending, internal stresses, etc. Internal stress could be caused, for instance, by fast quenching of thick-walled pipes.

The extrapolation of the results obtained with this method, to any kind of pipes, can be made only when strong internal stresses are not induced in the pipes.

As the conditions of use vary a great deal, it is important to carry out a preliminary determination of the chemical resistance of polyolefin pipes by means of simple, straightforward tests.

The purpose of this International Standard is to provide :

— a procedure;

— a standardized system for preliminary classification relating to the behaviour of pipes in relation to the chemical agents directly applicable to the transport of fluids in the absence of pressure.

If the pipes are to be used under stress, for example for transporting fluids under pressure, the method only allows incompatibilities between the fluid and the material to be detected; a "satisfactory" or "limited" result must be confirmed by subsequent tests according to a method under study in TC 138/SC 3, with the "corrosion factor" being determined under stress.

Some fluids may induce environmental stress-cracking effects.[1)]

1 Scope and field of application

1.1 This International Standard specifies a method to be used when carrying out a preliminary evaluation of the behaviour of polyolefin pipes in relation to the chemical fluids transported.

1.2 This standardized method of classification provides information on the suitability of pipes for transporting chemical fluids in the absence of pressure or stresses (earth loads, dynamic stresses, internal stresses, etc.).

1.3 A full procedure for carrying out the test is also reported in ISO 175, devoted to plastics in general, not in particular to polyolefin pipes.

2 References

ISO 175, *Plastics — Determination of the action of liquid chemicals, including water.*

ISO 527, *Plastics — Determination of tensile properties.*[2)]

ISO 1516, *Paints, varnishes, petroleum and related products — Flash/no flash test — Closed cup equilibrium method.*

ISO 3680, *Paints, varnishes, petroleum and related products — Flash/no flash test — Rapid equilibrium method.*

ISO 4451, *Polyethylene (PE) pipes and fittings — Determination of reference density of uncoloured and black polyethylene.*

ISO 6259, *Polyethylene (PE) pipes — Determination of tensile properties.*[3)]

1) For these cases, see ISO 4600 and ISO 4652.

2) At present at the stage of draft. (Revision of ISO/R 527-1966.)

3) At present at the stage of draft.

3 Symbols

The following symbols are used to indicate the behaviour of pipes in contact with chemical agents :

"S" : satisfactory resistance

The pipes can be used for applications where there is no pressure or other stress; for applications where there is pressure, the final evaluation shall be on the basis of a subsequent test under pressure.

"L" : limited resistance

The pipes can be used for applications where there is no pressure or other stress, but a certain amount of corrosion can be accepted; for applications where there is pressure, the final evaluation shall be on the basis of a subsequent test under pressure.

"NS" : non-satisfactory resistance

The pipes are severely attacked : they shall not be used for either pressure or non-pressure applications; there is no purpose in conducting tests under pressure as the results would certainly be unfavourable.

4 Principle of method

4.1 Standardized specimens (of the type used for tensile tests) are taken from pipes with normal wall thickness and made from the material to be tested.

4.2 The specimens are completely immersed in the chemical fluid being tested.

4.3 The immersion periods are standardized and chosen according to the change in mass of the specimens as a function of time.

4.4 The classification is based on the variation in certain properties of the specimens in the standardized tensile test when they have been immersed in the fluid for the standard times.

NOTE — Additional information is required when

— the pipes are permeable to the fluids transported;

— surface electrostatic charges can present a risk (fluids the flash point of which is less than 55 °C; the flash point can be determined according to ISO 1516 or ISO 3680);

— the immersion liquid can produce particular effects, such as stress cracking phenomena, which this method does not cover.

Section one : Test method

5 General test conditions

The general test conditions are those described in ISO 175 with the following more detailed specifications.

5.1 Test liquids

5.1.1 When information is required on the behaviour of a polyolefin pipe used to transport a specific liquid, this liquid shall normally be used.

5.1.2 The composition of industrial liquids is not, in general, absolutely constant; whenever possible, therefore, the test shall be carried out in defined chemical fluids used on their own or in mixtures and as representative as possible of the action of the products in question.

5.2 Test temperatures

Maintain the test liquid by suitable means at one of the temperatures in the table below.

NOTE — In the case of liquid having a boiling point below a temperature given in the table, the test must be carried out at the boiling point of the liquid.

5.3 Specimens

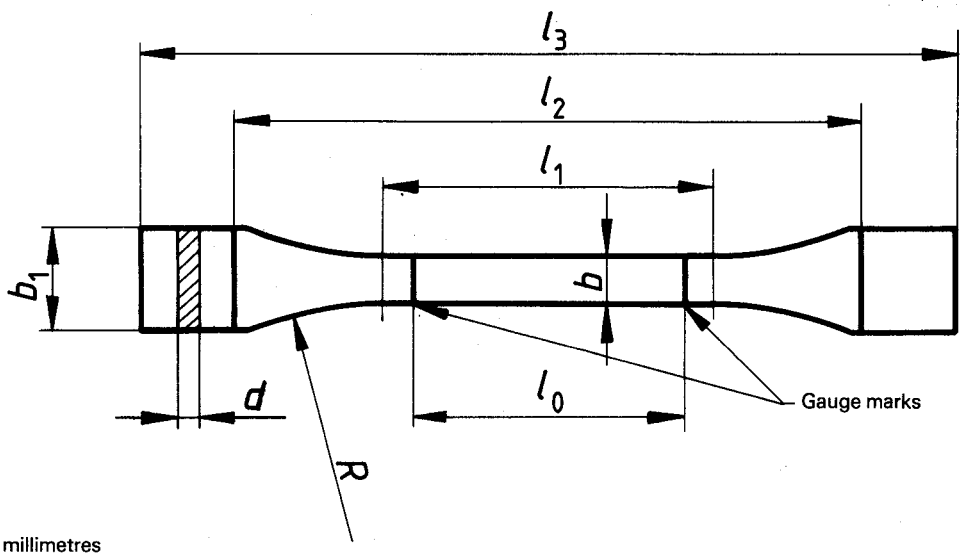
5.3.1 Type of specimen

The shape and dimensions of this test piece are given in figure 1.

This test piece is half the size of test piece type 1 specified in ISO 527.

Table — Test temperatures

Material \ Temperature, °C	23 ± 2	60 ± 2	80 ± 2	100 ± 2	120 ± 2
HDPE	x	x	x	x	—
LDPE	x	x	x	—	—
PP	x	—	x	x	x
PB	x	—	x	x	—



Dimensions in millimetres

l_3 Overall length, min. : 75

b_1 Width at ends : 10 ± 0,5

l_1 Length of narrow parallel portion : 30 ± 0,5

b Width of narrow parallel portion : 5 ± 0,5

R Radius, min. : 30

l_0 Distance between gauge marks : 25 ± 0,5

l_2 Initial distance between grips : 60 ± 5

d Thickness (see ISO 6259)

Figure 1 — Test piece

5.3.2 Number of specimens

The minimum number of specimens to be prepared is 20 for each test liquid at each temperature.

5.3.3 Obtaining specimens

The pipes used for taking specimens shall :

— have been extruded for at least 3 days [10 d in the case of polybutylene (PB)];

— conform to the general specifications for polyolefin pipes;

— have a thickness of 2,2 mm ± 0,3 mm.

Test specimens should be obtained in such a way that their axis is parallel to that of the pipe and should be taken regularly from around its circumference.

For pipes of small size (OD < 50 mm), open the pipe along a generatrix. For pipes of OD ⩾ 50 mm, divide the pipe into two equal strips, in accordance with ISO 6529.

In relation to those specimens, two procedures can be used :

a) procedure A : using non-annealed samples; it provides information on the suitability of pipes in non-pressurized systems.

b) procedure B : using annealed samples; this procedure can be used, as a supplementary test, for the characterization of the material. In this case, in order to develop a uniform crystalline structure which is reproducible, the samples (the opened pipe or the two sections as described above) are to be annealed as follows, in accordance with ISO 4451 :

1) place the samples in an oven at the temperature :

— for polypropylene (PP) : 150 °C

— for high density polyethylene (HDPE) : 120 °C

— for low density polyethylene (LDPE) and polybutylene (PB) : 100 °C

and maintain them at that temperature for 1 h.

2) allow the samples to cool from the annealing temperature at a constant rate of 5 K/h.

From the opened pipe or from each of the strips (in the case of procedure B preferably directly after removing from the oven), cut out the test specimens using a clean sharp edged punch free from notches, having the shape illustrated in figure 1 of ISO 6259. Make the cut by applying a uniform force to the surface of the inside wall of the pipe.

5.3.4 Storage of specimens before immersion test

Store the prepared specimens under normal conditions at approximately 23 °C and 50 % relative humidity.

The immersion test and the tests on the non-immersed specimens shall not be carried out until the specimens have been kept under these normal conditions for a minimum of 4 h.

6 Immersion test procedure

6.1 Equipment

See ISO 175.

6.2 Number and intended use of specimens

For each test liquid, at least 20 specimens should be prepared :

— one set of at least five specimens to determine the initial tensile properties;

— three sets of at least five specimens which will be immersed in the liquid to determine the tensile properties after immersion times t_1, t_2 and t_3 as defined in 6.4.

6.3 Steps to be taken before every immersion

Before every immersion, measure the width and thickness of the specimens in their calibrated section to the nearest 0,02 mm and mark the specimens clearly to prevent any confusion.

6.4 Duration of immersion tests

The immersed specimens are removed from the liquid for tensile tests after three consecutive immersion periods t_1, t_2 and t_3 chosen from the following :

7 d — 14 d — 28 d — 56 d — 112 d.

The initial time is determined by the appearance of a plateau in the curve showing the change in mass as a function of time (see clause 7) and shall not exceed 28 d, even if no plateau appears in the change in mass diagram before 28 d.

6.5 Quantities of liquid to be used

Generally speaking, (i.e., in the case of tubes not containing extractible products or products very prone to attack) the quantity of liquid to be used must be at least 4 ml/cm^2 of total surface of the specimens.

NOTE — The total area of an ISO 1/2 specimen 2,2 mm thick is approx. 15 cm^2.

Each sample shall be immersed in a minimum of 60 ml; this requires 0,9 l for a set of 15 specimens.

6.6 Positioning of specimens

As the specimens are identical, it is allowable to place several specimens in the same container as long as they do not touch each other :

— arrange for the surface of the specimen in contact with the walls of the container to be as small as possible, for

example by ensuring that one edge is resting on the bottom of the container and the other on the vertical wall, or by suspending them;

— according to the circumstances, cover or stopper the containers and place them in the thermostatically-controlled enclosure. If there is a possibility of light affecting the action of the liquid, it is recommended to operate either in darkness or under defined conditions of illumination.

6.7 Replacing the test liquid

During the test, stir the liquids once a day and replace the liquid every 7 d.

If the liquid is unstable (for example sodium hypochlorite), replace it more frequently.

6.8 Rinsing and drying of specimens

At the end of the immersion period, bring the specimens to ambient temperature, by transferring them into test liquid at the temperature of the laboratory; this reconditioning may last for 3 ± 1 h.

Remove the specimens from the liquid and :

— if the liquid is an acid, a base or an aqueous solution, rinse them rapidly in water and dry them with a filter paper or a cloth which does not shed fluff;

— if the liquid is a non-volatile organic liquid insoluble in water, rinse the specimens with an inert but volatile solvent such as petroleum ether or methanol and dry them rapidly;

— if the liquid is a volatile solvent, dry them rapidly.

7 Determination of the change in mass as a function of the immersion period

The main purpose of this determination is to determine the times after which the tensile tests on immersed specimens are to be conducted. It is carried out on one of the three sets of immersed specimens, each element of which must be clearly marked.

7.1 Procedure

7.1.1 Determine the mass m_1 of each specimen to the nearest milligram prior to immersion.

7.1.2 Select the test liquid(s) in accordance with 5.1 and the temperatures in accordance with 5.2.

7.1.3 Immerse the specimens as described in clause 6.

7.1.4 At the end of the chosen immersion period, place each rinsed and dried specimen in a tared weighing bottle, stopper it and determine the mass m_2 of the specimen after immersion to the nearest milligram. If the liquid used for the test is very volatile at ambient temperature, the period during which the specimen is exposed to the atmosphere shall not exceed 30 s.

7.1.5 Re-immerse the specimen in the test liquid as soon as weighing is completed.

7.2 Frequency of measurements

The change in mass should be determined on samples selected after immersion for 24 h, 3, 7, 14, 28 d and ultimately after immersion for 56 d and 112 d.

7.3 Calculation and expression of the percentage of change in mass

7.3.1 Calculate the percentage increase (or decrease) in mass ΔM for each specimen after immersion, using the formula :

$$\Delta M = \frac{m_2 - m_1}{m_1} \times 100$$

where

m_1 is the initial mass of the specimen (before immersion);

m_2 is the mass of the specimen after immersion.

7.3.2 Calculate the arithmetic mean of the results relating to the specimens from the same pipe sample and for the same period of immersion at the same temperature.

7.4 Graphical expression of the result

After each determination, it is recommended the results should be recorded in the form of a graph, giving ΔM as a function of the square root of the time, such as that shown in annex A.

7.5 Application of the results

The curve showing the variation in mass as a function of time can have various forms, the most common of which are shown diagrammatically in annex B.

When a curve of type 4 or 7 is obtained (shown below) having a plateau, it is possible to identify the time t_1 after which the subsequent change in mass is negligible. The tensile tests defined in clause 8 are then determined using t_1 days as the initial immersion time, which depends on the time to saturation, and can commence at either 7, 14 or 28 d.

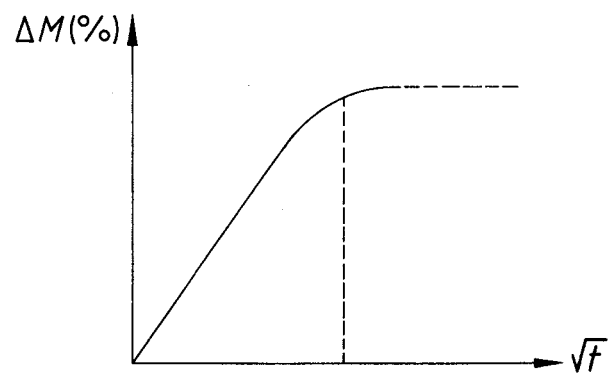


Figure 2 — Curve No. 4

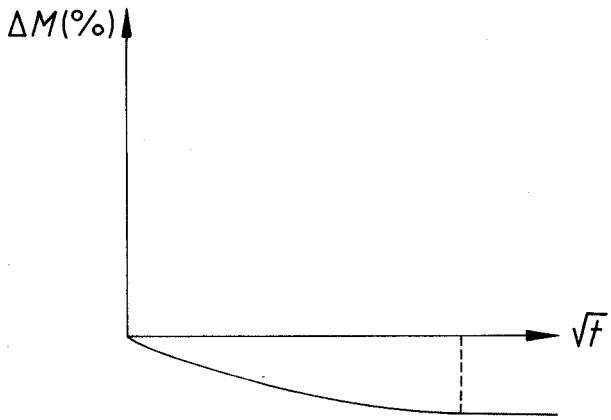


Figure 3 — Curve No. 7

If the curve obtained is one of the alternative types, 1, 2, 3, 5 or 6, the time t_1 is always 28 d.

8 Determination of changes in tensile properties

8.1 Apparatus

Use a tensile-testing machine which permits the loads applied and the corresponding elongations, which shall be measured with an extensiometer, to be determined.

8.2 Conditioning of specimens

The determination of tensile properties is carried out on sets of at least five specimens which have been treated in the same way; either no immersion in the case of initial properties or immersion in the same fluid and for the same period at the same temperature.

When the immersion tests have been carried out at temperatures above 23 °C, immerse the test specimens for 3 h at 23 ± 2 °C in the same test liquid before being submitted to tensile tests.

The specimens shall be tested not later than 2 h after their removal from the cold liquid. During this period they shall be kept at 23 ± 2 °C.

However, if the liquid used for the test is very volatile at ambient temperature, the test shall be carried out within 2 to 3 min after the removal of the specimen from the cold liquid.

8.3 Speed of testing

The speed of testing, that is the velocity of separation of the grips of the apparatus during measurement, shall be 100 ± 10 mm/min, irrespective of the type of polyolefin.

8.4 Procedure

Carry out the test at 23 ± 2 °C.

Place the specimen in the grips of the machine.

Set the extension indicator with a distance of 25 ± 0,5 mm between its grips.

Set the speed of testing at 100 ± 10 mm/min.

Start the machine.

Note or record the following details :

a) the existence of a yield point causing necking;

b) the value of the load at the yield point F_y or F_{yo} (where the suffix o relates to the original value, that is before immersion);

c) the elongation at the yield point E_y or E_{yo};

d) the load at break F_b and the elongation at break E_b if there is wide scatter on the values of elongation at yield.

NOTE — The width and thickness of the specimen in the calibrated section are to be measured to an accuracy of 0,02 mm immediately before immersion (see 6.2).

The above information can be obtained by means of automatically-recorded stress curves or by direct observation.

8.5 Calculation of stresses at yield and at break

For each specimen, calculate the stresses using the following formulae :

$$\sigma_y = \frac{F_y}{w \times t}$$

and

$$\sigma_b = \frac{F_b}{w \times t}$$

where

σ_y is the stress at yield, in newtons per square millimetre;

σ_b is the stress at break, in newtons per square millimetre;

F_y is the load at yield, in newtons;

F_b is the load at break, in newtons;

w is the initial width of specimen, in millimetres;

t is the initial thickness of specimen, in millimetres.

Express the results to three significant figures.

8.6 Calculation of elongations at yield and at break

For each specimen, calculate the elongations using the following formulae :

$$E_y = \frac{L_y - L_o}{L_o} \times 100$$

and

$$E_b = \frac{L_b - L_o}{L_o} \times 100$$

where

E_y is the elongation at yield, expressed as a percentage;

E_b is the elongation at break, expressed as a percentage;

L_y is the length of the calibrated section of the specimen at yield, in millimetres;

L_o is the initial length of the calibrated section of the specimen, in millimetres;

L_b is the length of the calibrated section of the specimen at break, in millimetres.

Express the results to three significant figures.