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Polyolefin pipes for the conveyance of fluids — Determination of resistance to crack propagation — Test method for slow crack growth on notched pipes

Tubes en polyoléfines pour le transport des fluides — Détermination de la résistance à la propagation de la fissure — Méthode d'essai de la propagation lente de la fissure d'un tube entaillé (essai d'entaille)



Reference number ISO 13479:2022(E)



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Foreword

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

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

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, Subcommittee SC 5, *General properties of pipes, fittings and valves of plastic materials and their accessories -- Test methods and basic specifications,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 155, *Plastics piping systems and ducting systems*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 13479:2009), which has been technically revised.

The main changes are as follows:

- warnings have been added to follow the method of test piece preparation and the test procedure because of the influence on the result;
- a maximum notch radius has been specified;
- in case of premature failure, alternative test pressures and times for PE 80 and PE 100 have been added to allow retesting at a lower pressure for a longer time;
- an accelerated method by testing with an external detergent has been added.

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

Polyolefin pipes for the conveyance of fluids — Determination of resistance to crack propagation — Test method for slow crack growth on notched pipes

1 Scope

This document specifies a test method for determining the resistance to slow crack growth of polyolefin pipes, expressed in terms of time to failure in a hydrostatic pressure test on a pipe with machined longitudinal notches in the outside surface. The test is applicable to pipes of wall thickness greater than 5 mm.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 161-1, Thermoplastics pipes for the conveyance of fluids — Nominal outside diameters and nominal pressures — Part 1: Metric series

ISO 1167-1, Thermoplastics pipes, fittings and assemblies for the conveyance of fluids — Determination of the resistance to internal pressure — Part 1: General method

ISO 1167-2, Thermoplastics pipes, fittings and assemblies for the conveyance of fluids — Determination of the resistance to internal pressure — Part 2: Preparation of pipe test pieces

ISO 3126, Plastics piping systems — Plastics components — Determination of dimensions

ISO 11922-1, Thermoplastics pipes for the conveyance of fluids — Dimensions and tolerances — Part 1: Metric series

ISO 15510, Stainless steels — Chemical composition

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 161-1 and ISO 11922-1 and the following apply.

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

- ISO Online browsing platform: available at <u>https://www.iso.org/obp</u>
- IEC Electropedia: available at https://www.electropedia.org/

3.1 Terms related to geometrical dimensions

3.1.1

nominal outside diameter

 $d_{\rm n}$ specified outside diameter assigned to a nominal size DN/OD

Note 1 to entry: Nominal outside diameter is expressed in millimetres.

3.1.2

mean outside diameter

d_{em}

value of the measurement of the outer circumference of the pipe or spigot end of a fitting in any crosssection divided by π (= 3,142), rounded to the next greater 0,1 mm

3.1.3

minimum wall thickness

 e_{\min}

minimum value for the wall thickness at any point around the circumference of a component, as specified

3.1.4

standard dimension ratio

SDR

numerical designation of a pipe series, which is a convenient round number, approximately equal to the dimension ratio of the nominal outside diameter, d_n , and the nominal wall thickness, e_n

3.1.5

pipe series number for pipe designation

Note 1 to entry: Pipe series values are defined according to ISO 4065.

Note 2 to entry: The relationship between the pipe series, S, and the standard dimension ratio, SDR, is given in ISO 4065 as follows.

$$S = \frac{SDR - 1}{2}$$

3.1.6 ligament thickness

 $\delta_{
m lg}$

value of the measurement or calculation of the remaining ligament after machining of the notch

3.1.7

notch depth *h*

value of the depth of the notch after machining, measured or calculated

3.1.8

notch length

 $l_{\rm n}$ value of the length of the notch

3.1.9

width of machined surface of notch

b_s

value of the width of the machined surface of the notch

3.2 Terms related to machining of notches

3.2.1

climb milling

milling in which the cutting motion of the tool is in the same direction as the feeding direction of the component being milled

Note 1 to entry: This is also referred to as 'down milling'.

3.2.2 revolution of the cutter *r*

value used as a basis for the cutting rate

4 Symbols and abbreviation

4.1 Symbols

For the purposes of this document, the following symbols apply.

- $b_{\rm s}$ width of machined surface of the notch
- $d_{\rm em}$ mean outside diameter
- *d*_n nominal outside diameter
- *e* wall thickness (at any point) of a pipe
- *e*_m mean wall thickness
- e_{\max} maximum wall thickness (at any point) of a pipe
- e_{\min} minimum wall thickness (at any point) of a pipe
- *h* notch depth
- *l*_n notch length
- *p* test pressure
- *r* revolution of the cutter
- $\delta_{
 m lg}$ ligament thickness
- σ hydrostatic stress, in megapascals

4.2 Abbreviated terms

- ANPT accelerated notch pipe test
- PE polyethylene
- RC raised crack resistance
- S pipe series
- SDR standard dimension ratio

5 Principle

A length of pipe with four machined longitudinal external notches is subject to a hydrostatic pressure test whilst immersed in a water tank at 80 °C in accordance with ISO 1167-1 and ISO 1167-2. The time to failure or test period is recorded.

NOTE 1 It is assumed that the following test parameters are set by the standard or specification making reference to this test method:

a) the number of test pieces, if applicable (see 7.5);

- b) the test pressure (see <u>9.1</u>);
- c) the test period (see <u>9.1</u>).

To accelerate the test, the pipe with external machined notches is immersed in a tank containing a detergent, for example $Arkopal^{(B)} N100^{1}$, in accordance with <u>Annex D</u>.

6 Apparatus

6.1 Pipe pressure-testing equipment, as specified in ISO 1167-1.

NOTE Ideally when testing notched pipes, it is recommended to use a single test station. Furthermore, it is recommended to use automated shut off equipment for each individual pipe when testing multiple pipes on a manifold. Otherwise, when a pipe fails, the other pipes are disturbed, and re-pressurizing can accelerate any crack growth present in the notches.

6.2 Notch machining equipment, for example a milling machine with a horizontal mandrel rigidly fixed to the bed to enable the pipe to be securely clamped to give a straight test piece.

Alternatively, the pipe to be notched can also be fixed from the outside with suitable clamps to keep it in a stable position to avoid vibrations during the notching process.

The milling cutter mounted on a horizontal arbor shall be a 60° double equal angle V-cutter with a pointed tip, having a calculated cutting rate of (0,010 ± 0,002) (mm/r)/tooth (see example).

It is important that the cutting rate is within the specified range, otherwise the results will not be valid.

EXAMPLE A cutter with 20 teeth rotating at 700 r/min, traversed at a speed of 150 mm/min, has a calculated cutting rate of $150/(20 \times 700) = 0.011 \text{ (mm/r)}/tooth$.

Vibration of the cutter or the machine bed can affect the radius formed at the bottom of the notch and shall be minimized.

The milling cutter shall be carefully protected against damage. The cutter shall be subject to a runningin treatment amounting to 10 m of notching at the specified cutting rate, prior to its first use for the preparation of test pieces. It shall not be used for any other material or purpose and shall be replaced after 500 m of notching.

The cutter shall be checked for damage or wear after not more than 100 m of cutting. The cutter teeth shall be compared with a new cutter by examination with a microscope using a magnification of 10 to 20 times. If there is any evidence of damage or wear it shall be replaced.

The quality of the cutter and machining process can be checked by carrying out notching of a sample and visually checking the notch tip radius after cutting the cross-section of the pipe. This shall be done after installation of a new cutter.

6.3 End caps, type A in accordance with ISO 1167-1.

7 Test piece preparation

7.1 General

Prior to any measurements the test piece shall be conditioned at (23 ± 2) °C for at least 4 h.

¹⁾ Arkopal® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO. See <u>Clause D.3</u>, NOTE 2 concerning ongoing research to find alternative stress cracking media to replace nonylphenol ethoxylate types providing accepted correlation has been developed.

7.2 Test pieces

Each test piece shall comprise a length of pipe sufficient to give a minimum free length of pipe of $(3d_n \pm 5)$ mm between the end caps, when fitted for pressure testing in accordance with ISO 1167-2, where d_n is the nominal outside diameter of the pipe. For pipes with a nominal outside diameter $d_n > 315$ mm, a minimum free length of $(3 d_n \pm 5)$ mm shall be used where practical; otherwise a minimum free length of ≥ 1000 mm shall be used.

NOTE It is possible that the use of pipes less than 3 d_n and notch lengths less than or greater than the nominal outside diameter will affect the results.

7.3 Notch location and measurement of dimensions

Positions shall be marked for machining four notches equally spaced around the pipe circumference (see Figure 1). Measure the mean outside diameter, d_{em} , of the test pipe and the wall thickness of the pipe in the centre of the pipe at each notch position in accordance with ISO 3126.

7.4 Machining the notches

7.4.1 If the wall thickness of the test piece is greater than 50 mm, the material shall be machined with a slot drill of 15 mm to 20 mm diameter to leave approximately 10 mm to be removed by the V-cutter, used in accordance with <u>7.4.2</u>. Machining of notches shall not take place within 24 h of production of the pipe.

7.4.2 Each notch shall be machined by climb milling (see Figure 2), to such a depth as to produce a pipe wall ligament thickness of between 0,78 and 0,82 times the minimum wall thickness, e_{min} , as specified in ISO 11922-1, for the diameter and pressure series of the pipe as shown in Table A.1. The ends of each notch shall be aligned circumferentially as shown in Figure 1 and Figure 2. It is important that the climb milling technique is used, otherwise the results will not be valid.

Vibration of the cutter or machine bed can affect the radius formed at the bottom of the notch and shall be minimized.

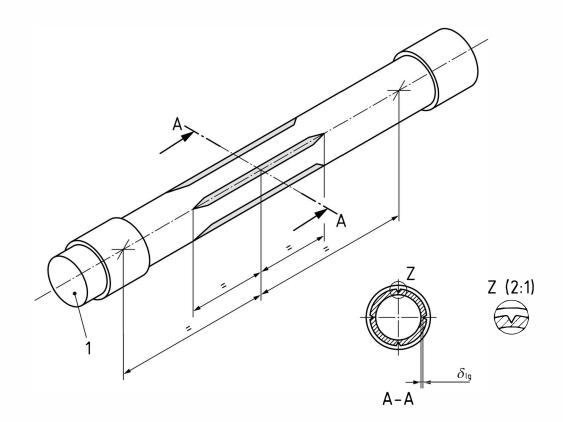
The notch radius shall not be greater than 100 μ m when measured in accordance with the method described in <u>Annex E</u> and shall be checked at regular intervals of not more than 100 m of cutting.

The length of each notch, at full depth, shall be equal to the pipe nominal outside diameter ± 1 mm. For pipes greater than 315 mm in diameter with a free length of pipe of less than $(3 d_n \pm 5)$ mm, the length of each notch, at full depth, shall be equal to the free length minus (500 ± 1) mm, in accordance with 7.2.

NOTE To achieve a remaining ligament within the required tolerance range, it is advisable to aim for a remaining ligament at the maximum of the tolerance range. This is because the pipe wall can move due to the release of residual stresses, resulting in a deeper than anticipated notch.

7.4.3 Measure and record the depth of each notch and the ligament thickness, δ_{lg} , by contact mechanical measurement or non-contact means.

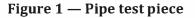
NOTE An example of a non-contact method is given in Reference [4] of the Bibliography.

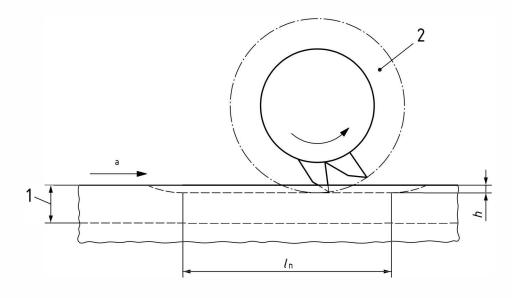


Key

1 end cap

 $\delta_{\rm lg}$ ligament thickness: 0,78 to 0,82 times minimum thickness specified by the product standard for the pipe being tested, in millimetres





Key

- 1 pipe wall
- 2 60° double equal angle V-cutter
- *h* notch depth, in millimetres
- $l_{\rm n}$ notch length (1 × $d_{\rm n}$) centred on test piece
- ^a Direction.

Figure 2 — Notching method

7.5 Number of test pieces

Prepare a minimum of three test pieces, unless specified otherwise in the referring standard or specification.

8 Conditioning

The test pieces shall be filled with water, immersed in a water tank at 80 $^{\circ}$ C and allowed to condition for 24 h ± 1 h for wall thickness up to 25 mm and 48 h ± 1 h for greater wall thickness.

9 Procedure

9.1 Hydrostatic-pressure testing

9.1.1 An internal pressure test in accordance with ISO 1167-1 at a test temperature of 80 $^{\circ}$ C shall be carried out on the prepared test piece by applying and maintaining the pressure specified by the referring standard.

9.1.2 Connect the test piece(s) to the pressurizing equipment and bleed off the air. After conditioning in accordance with <u>Clause 8</u>, progressively and smoothly apply the test pressure, in the shortest time practicable between 30 s and 1 h, depending upon the size of the test piece and the capability of the pressurizing equipment.

NOTE Shock loading by rapid increase of pressure can create crack tip blunting that will affect the test result.

9.1.3 Maintain the pressure until either the test piece ruptures or the time specified by the referring standard has elapsed, whichever occurs first. Record the time under pressure to the nearest hour. In the case of failure, record the location of the failure for each test piece.

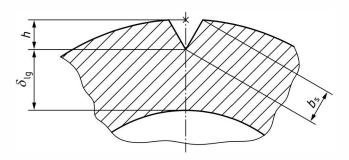
<u>Table B.1</u> gives recommended applicable pressure levels for polyethylene pipes, dependent on material type and pipe series.

9.1.4 If the sample fails prematurely before the specified requirement, a retest can be performed at a selected lower pressure for a longer time, if permitted by the referring standard. <u>Annex C</u> gives this information for polyethylene.

9.2 Ligament thickness measurement after testing

Measurement after testing shall be carried out if premature failure has occurred, or for the purpose of verifying not ch depth measurement according to 7.4.3, using the following method. If the remaining ligament is found to be outside the tolerance range, the test result shall be discarded. A retest shall be performed using the same conditions and requirements.

On completion of the pressure test, remove the test piece from the water tank and allow to cool to ambient temperature. Cut a section of pipe out from around the position of each notch. Open up the notch to give clear access to one of the machined surfaces of the notch. Measure the width of the machined surface of the notch, $b_{s'}$ to an accuracy of ±0,1 mm with a microscope or equivalent means, as shown in Figure 3. If required by the referring standard, measure the depth of penetration of the crack.



Key

 $b_{\rm s}$ width of machined surface of notch

h notch depth

 $\delta_{
m lg}$ ligament thickness

Figure 3 — Measurement to calculate notch depth

Calculate the notch depth, *h*, in millimetres, using Formula (1):

$$h = 0.5 \left[d_{\rm em} - \sqrt{\left(d_{\rm em}^2 - b_{\rm s}^2 \right)} \right] + 0.866 \, b_{\rm s} \tag{1}$$

where

*b*_s is the width of machined surface of the notch, in millimetres;

 $d_{\rm em}$ is the measured mean pipe outside diameter, in millimetres.

Calculate the ligament thickness, δ_{lg} , from the notch depth and the individual average wall thickness alongside each notch position. Record the values obtained.

NOTE An alternative method is detailed in Reference [4] of the Bibliography.

10 Test report

The test report shall include the following information:

- a) reference to this document, i.e. ISO 13479:2022, and to the referring standard or specification if applicable;
- b) all details necessary for complete identification of the pipe (manufacturer, type of pipe and production date);
- c) the cutter size and number of teeth;
- d) the cutter speed, in revolutions per minute and the traverse speed, in millimetres per minute;
- e) the mean pipe outside diameter, in millimetres, and the pipe series or SDR;
- f) the notch depth and ligament thickness for each notch;
- g) the location of any failed notch, and failure mode;
- h) the test pressure, and selected retest pressure if applicable;
- i) the test temperature;
- j) the time under pressure or the time to failure, in hours, as applicable;

- k) the details of any factors which could have affected the results, such as any incidents or any operations not specified in this document;
- l) any unusual features observed;
- m) the date of the test.

Annex A

(normative)

Ligament thicknesses

See <u>Table A.1</u> for remaining ligament thicknesses based on the pipe series of the pipe to be tested.

Nominal outside		PR 6 2,5		R 7,4 3,2		R 9 4		R 11 5	SDR S (-	SDF S	8	S 8	17,6 3,3	SDF S 1		SDR S 12			R 33 16	SDF S 2	
diameter		Ligament thickness, δ_{lg}																				
d _n	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.
32	4,2	4,4																				
40	5,2	5,5	4,3	4,5																		
50	6,5	6,8	5,4	5,7	4,4	4,6																
63	8,2	8,6	6,7	7,1	5,5	5,8	4,5	4,8														
75	9,8	10,3	8,0	8,4	6,5	6,9	5,3	5,6	4,3	4,5												
90	11,7	12,3	9,6	10,1	7,9	8,3	6,4	6,7	5,1	5,4	4,2	4,4	4,0	4,2								
110	14,3	15,0	11,8	12,4	9,6	10,1	7,8	8,2	6,3	6,6	5,1	5,4	4,9	5,2	4,1	4,3						
125	16,2	17,1	13,3	14,0	10,9	11,5	8,9	9,3	7,2	7,5	5,8	6,1	5,5	5,8	4,7	4,9						
140	18,2	19,1	15,0	15,7	12,2	12,9	9,9	10,4	8,0	8,4	6,5	6,8	6,2	6,6	5,2	5,5	4,2	4,4				
160	20,7	21,8	17,1	18,0	14,0	14,7	11,4	12,0	9,2	9,7	7,4	7,8	7,1	7,5	6,0	6,3	4,8	5,1				
180	23,3	24,5	19,2	20,2	15,7	16,5	12,8	13,4	10,4	10,9	8,3	8,8	8,0	8,4	6,7	7,1	5,4	5,7	4,3	4,5		
200	25,9	27,2	21,4	22,5	17,5	18,4	14,2	14,9	11,5	12,1	9,3	9,8	8,9	9,3	7,5	7,9	6,0	6,3	4,8	5,1		
225	29,2	30,7	24,0	25,3	19,6	20,6	16,0	16,8	12,9	13,6	10,5	11,0	10,0	10,5	8,4	8,9	6,7	7,1	5,4	5,7	4,3	4,5
250	32,4	34,0	26,7	28,0	21,8	22,9	17,7	18,6	14,4	15,1	11,5	12,1	11,1	11,6	9,3	9,8	7,5	7,9	6,0	6,3	4,8	5,0
280	36,3	38,1	29,9	31,4	24,3	2 <i>5,</i> 6	19,8	20,8	16,1	16,9	12,9	13,6	12,4	13,0	10,5	11,0	8,3	8,8	6,7	7,1	5,4	5,7
315	40,8	42,9	33,6	35,3	27,3	28,7	22,3	23,5	18,2	19,1	14,6	15,3	14,0	14,7	11,7	12,3	9,4	9,9	7,6	8,0	6,0	6,3
355	46,0	48,4	37,8	39,8	30,8	32,4	25,2	26,5	20,4	21,4	16,5	17,3	15,8	16,6	13,2	13,9	10,6	11,2	8,5	8,9	6,8	7,1
400			42,7	44,9	34,7	36,5	28,4	29,8	22,9	24,1	18,5	19,4	17,8	18,7	14,9	15,7	11,9	12,5	9,6	10,1	7,6	8,0
450			48,1	50,6	39,0	41,0	31,9	33,5	25,8	27,1	20,8	21,9	19,9	21,0	16,8	17,6	13,4	14,1	10,8	11,3	8,6	9,0
500					43,4	45,6	35,5	37,3	28,7	30,2	23,1	24,3	22,2	23,3	18,6	19,6	14,9	15,7	11,9	12,5	9,5	10,0
560							39,7	41,7	32,1	33,8	25,9	27,2	24,9	26,2	20,8	21,9	16,7	17,5	13,4	14,1	10,7	11,2
630							44,7	47,0	36,2	38,0	29,1	30,6	27,9	29,4	23,4	24,6	18,8	19,8	15,1	15,8	12,0	12,6
710									40,8	42,9	32,8	34,5	31,4	33,0	26,4	27,8	21,2	22,3	17,0	17,9	13,6	14,3
800									45,9	48,3	37,0	38,9	35,3	37,1	29,7	31,2	23,9	25,1	19,1	20,1	15,3	16,1

Table A.1 — Remaining ligament thicknesses for pipe series

Dimensions in millimetres

Table A.1 (continued)																						
Nominal outside	1	R 6 2,5	S DF S 3	R 7,4 3,2	S D S	R 9 4	1	R 11 5	1	13,6 6,3	S D F S		SDR S 8	. 17,6 3,3	S DF S 1		S DR S 1			R 33 16	SDF S 1	R 41 20
diameter	diameter Ligament thickness, δ_{lg}																					
d _n	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.	min.	max.
900											41,7	43,9	39,8	41,8	33,5	35,2	27,1	28,5	21,5	22,6	17,2	18,0
1 000											46,3	48,6	44,1	46,4	37,2	39,1	30,0	31,6	23,9	25,1	19,0	20,0
1 200															44,6	46,9	36,0	37,9	28,4	29,8	22,8	24,0
1 400																	42,0	44,2	33,1	34,8	26,7	28,0
1 600																	48,0	50,4	37,8	39,8	30,5	32,1

12

Annex B (informative)

Test-pressure levels for polyethylene

For a notch test for slow crack growth in polyethylene (PE) pipe, at the test temperature of 80 °C the recommended applicable pressure levels depend on the material type and pipe series of test pipe as given in <u>Table B.1</u>. The recommended requirements are given in <u>Annex C</u>.

NOTE This test can be applicable to other thermoplastics, but test parameters would need to be developed.

Standard	S series	Test pressure , p bar					
dimension ratio (SDR)							
		PE 80	PE 100				
41	20	2	2,3				
33	16	2,5	2,88				
26	12,5	3,2	3,68				
21	10	4	4,6				
17,6	8,3	4,82	5,54				
17	8	5	5,75				
13,6	6,3	6,35	7,3				
11	5	8	9,2				
9	4	10	11,5				
7,4	3,2	12,5	14,38				
6	2,5	16	18,4				

Table B.1 — Test-pressure levels

NOTE 1 These pressure levels are calculated to give nominal plain-pipe hydrostatic stress levels of 4,0 MPa in PE 80 materials and 4,6 MPa in PE 100 materials using Formula (B.1):

$p = \frac{1}{2}$	$\frac{10\sigma}{S}$ or $p = \frac{20\sigma}{R_{\rm SD} - 1}$	(B.1)
where		
σ	is the hydrostatic stress, in megapascals;	
S	is the pipe series, S;	
R _{SD}	is the standard dimension ratio, SDR.	
NOTE 2	1 bar = 0,1 MPa = 10^5 Pa; 1 MPa = 1 N/mm ² .	

Annex C (informative)

Recommended requirements for polyethylene

C.1 Requirement for polyethylene

The recommended requirement for PE 80 and PE 100 pipes is \geq 500 h at a test temperature of 80 °C for test pressures given in <u>Table B.1</u>, or as defined by the referring standard.

C.2 Premature failure

In case of premature failure in tests for an SDR 11 PE 100 pipe at 9,2 bar pressure or an SDR 11 PE 80 pipe at 8,0 bar pressure, the pipe can be retested using the test parameters given in <u>Table C.1</u>.

PE 1	100	PE 80						
Pressure	Time	Pressure	Time					
Bar	h	Bar	h					
8,8	775	7,4	785					
8,4 1 225 6,8 1 275								
NOTE 1 bar = $0.1 \text{ MPa} = 10^5 \text{ Pa}$; 1 MPa = 1 N/mm^2 .								

Table C.1 — Test parameters for retest of SDR 11 PE pipes in case of premature failure

For pipes in other SDRs, the formula given in <u>Table B.1</u> is used to calculate the test pressure.

Annex D

(normative)

Test procedure for the accelerated notched pipe test (ANPT) for PE 100-RC pipes

D.1 General

For materials with high resistance to slow crack growth, such as PE 100-RC, the normal procedure of carrying out the pressure test in a water bath at 80 °C is expected to result in a test time in excess of one year, using the test parameters specified in this document.

To accelerate the test an external detergent solution in contact with the notches instead of water as the outer media is used to reduce the time to failure.

This annex specifies the equipment, preparation of the detergent solution, procedure and the precautions to be taken to ensure reproducibility and safety.

NOTE A combination of this test with a detergent solution of 2 % Arkopal[®] N100²) in demineralized water as the outer media, as defined for the ISO 16770 Full-Notch Creep Test^[2], is specified. The detergent solution parameters including ageing behaviour, circulation, and oxygen content is controlled. This results in brittle failure in a relatively short time even for PE 100-RC materials ^[5], ^[6].

D.2 Apparatus

Pressure test equipment with a tank to immerse the test pipes similar to that used for testing with water is required.

Care needs to be taken to check that all materials in contact with the detergent solution do not interact with it and change the activity, for example materials used for sealing. It is recommended to use stainless steel (e.g. 4571-316-35-I, 1,440 4 or 1,457 1 in accordance with ISO 15510) for the test bath, pumping device, and end caps for the pipe test piece.

To ensure homogeneity of the detergent solution it is necessary to have continuous circulation in the tank. It is recommended to extract the detergent solution at several positions at the base of the bath, and to pump it back in at about two thirds of the bath height to create flow to prevent sedimentation and segregation of the detergent solution and to maintain a consistent temperature distribution in the tank. It is recommended to seal the test tank with a lid to reduce evaporation and changes in concentration of the solution.

It is highly recommended to dedicate a test tank for testing with detergents, and to isolate this test tank to ensure that no contamination of other water tanks or equipment can occur.

D.3 Preparation of detergent solution

A non-ionic neutral type nonylphenol ethoxylate detergent according to CAS No. 9016-45-9 shall be used with an n-value of 10. The general formula of this type of detergent is shown in Figure D.1.

²⁾ Arkopal® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO. See <u>Clause D.3</u>, NOTE 2 concerning ongoing research to find alternative stress cracking media to replace nonylphenol ethoxylate types providing accepted correlation has been developed.

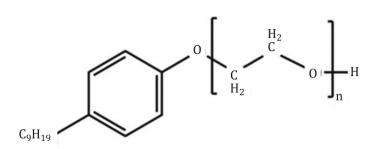


Figure D.1 — Formula of nonylphenol ethoxylate detergents (CAS No. 9016-45-9)

NOTE 1 An example of this commercially available detergent is Arkopal[®] N100³). This test environment is suitable for use in testing at elevated temperatures and is sufficiently aggressive to produce failures in reasonable times for PE 100-RC materials.

NOTE 2 Other surface-active test environments can be used as recommended in ISO 16770, if specified by referring standards, or agreed upon between interested parties. Research is ongoing to find alternative stress cracking media to replace nonylphenol ethoxylate types providing accepted correlation has been developed.

Using demineralized water, a sufficient quantity of solution shall be prepared, of a concentration equivalent to $(2 \pm 0,1)$ % mass fraction of detergent. A complete immersion of the test specimens in the detergent solution shall be ensured. To compensate for evaporated water, only demineralized water shall be added to re-establish the initial solution level. The solution shall be prepared by adding the detergent to the water heated to 80 °C.

D.4 Activity (ageing behaviour) of the detergent solution

The solution shall be aged for a minimum of 14 days at the test temperature before use to ensure consistent results. A shorter ageing time can be used if consistent solution activity is demonstrated using a test on a reference material. After a maximum of 2 000 h use, the solution shall be renewed due to acidification of the detergent occurring at 80 °C, and in contact with air. Forward planning of testing is required to ensure that tests do not exceed maximum use of the solution.

Renewing the solution during testing shall be avoided, to limit the disturbance of the un-failed test specimens.

To maintain consistent bath conditions, the activity of the detergent solution shall be monitored by a suitable technique, for example specimens of a reference material can be tested in the solution to verify that there is no change in activity.

For new test equipment it is recommended to carry out the test procedure with a reference material at least once to get information about the activity and stability (ageing behaviour) of the detergent solution, which is dependent on the test set up.

D.5 Preparation of pipe test pieces

The pipe test pieces with machined notches shall be prepared in accordance with <u>Clause 6</u>.

³⁾ Arkopal® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO. See <u>Clause D.3</u>, NOTE 2 concerning ongoing research to find alternative stress cracking media to replace nonylphenol ethoxylate types providing accepted correlation has been developed.

D.6 Conditioning

The pipe test pieces shall be filled with water, immersed in the tank containing the detergent solution at 80 °C, and allowed to condition for 24 h \pm 1 h for wall thickness up to 25 mm and 48 h \pm 1 h for greater wall thickness.

D.7 Test procedure

Following the conditioning in the test tank, the pipe shall be pressurised in accordance with 9.1.2. Following completion of the pressure test the notch ligament depth can be checked in accordance with 9.2, if required.

D.8 Test report

The information required in accordance with <u>Clause 10</u> shall be recorded. In addition, the following information shall be added:

- a) reference to this annex;
- b) details of the detergent solution, i.e. CAS number, type, trade name, concentration and age of solution at the start of the test

Annex E

(normative)

Method of measuring notch radius

The following steps shall be taken to measure the radius of the notch in a pipe:

- a) Machine 4 notches in a 110 mm SDR 11 pipe following <u>subclause 7.4</u> of this document.
- b) Cut the pipe perpendicular to its axis from the midpoint of the notches.
- c) Cut a ring of minimum 20 mm width from the notched end of the pipe.
- d) Cut a minimum 10 mm circumferential section from the pipe ring where one of the notches is in the middle of the cut section.
- e) Use a microtome to remove thin layers from the notch cross-section until a flat and smooth surface is obtained. The microtome cutting direction shall be from the pipe outside to the notch tip to prevent debris forming in the tip.
- f) Look at the microtome sliced cross-section of the notch using a microscope with a suitable magnification, acquire a cross-section image with a scale bar embedded.
- g) Using image processing software, put lines on each notch edges and measure the notch angle.
- h) Using image processing software put a circle close to the notch tip where the notch angle lines have a tangent to the circle. Measure the radius of the circle.
- i) Repeat these steps for other notches if necessary.

Bibliography

- [1] ISO 6108⁴), Double equal angle cutters with plain bore and key drive
- [2] ISO 16770, Plastics Determination of environmental stress cracking (ESC) of polyethylene Full-notch creep test (FNCT)
- [3] Accelerated pipe test methods to evaluate PE 100-RC materials Possibilities for ISO standardisation, Kratochvilla T, Eremiasch R, Bruckner C, TGM Austria, Proceedings of the 19th Plastic Pipes Conference PPXIX, September 2018
- [4] The notched cylindrical bars under constant load test" (NCBT) for assessing the resistance to crack initiation and to slow crack growth of PE100 and PE100 RC, Gueugnaut D, Boujlal A, Lopitaux A, Bouaffre R, proceedings of the 17th Plastics Pipes Conference PPXVII, September 2014.
- [5] Accelerated Notch Pipe Test (ANPT) on pipes made of PE 80, PE 100 and PE 100-RC, TGM Austria, (TGM VA KU 25 550/8), 6th December 2019, see <u>www.pe100plus.com</u>.
- [6] Interlaboratory comparison Accelerated Notch Pipe test (ANPT) ISO 13479, KIWA The Netherlands, 22nd October 2020 (LC 18841-2a), see <u>www.pe100plus.com</u>.

⁴⁾ Withdrawn.

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