

Designation: F1473 - 16 F1473 - 18

Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resins¹

This standard is issued under the fixed designation F1473; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method determines the resistance of polyethylene materials to slow crack growth under conditions specified within.

Note 1—This test method is known as PENT (Pennsylvania Notch Test) test.

- 1.2 The test is generally performed at 80°C and at 2.4 MPa, but may also be done at temperatures below 80°C and with other stresses low enough to preclude ductile failure and thereby eventually induce brittle type of failure. Generally, polyethylenes will ultimately fail in a brittle manner by slow crack growth at 80°C if the stress is below 2.4 MPa.
- 1.3 The test method is for specimens cut from compression molded plaques.² See Appendix X1 for information relating to specimens from pipe.
 - 1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety safety, health, and health environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.6 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:³

D1600 Terminology for Abbreviated Terms Relating to Plastics

D4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets

D5596 Test Method For Microscopic Evaluation of the Dispersion of Carbon Black in Polyolefin Geosynthetics

F412 Terminology Relating to Plastic Piping Systems

2.2 ISO Standard:⁴

ISO 18553 Method for the assessment of the degree of pigment or carbon black dispersion in polyolefin pipes, fittings and compounds

3. Terminology

- 3.1 Definitions are in accordance with Terminology F412. Abbreviations are in accordance with Terminology D1600, unless otherwise indicated.
 - 3.2 Definitions:
- 3.2.1 *brittle failure*—a pipe failure mode which exhibits no visible (to the naked eye) permanent material deformation (stretching, elongation, or necking down) in the area of the break (Terminology F412).
 - 3.3 Definitions of Terms Specific to This Standard:

¹ This test method is under the jurisdiction of ASTM Committee F17 on Plastic Piping Systems and is the direct responsibility of Subcommittee F17.40 on Test Methods. Current edition approved Aug. 15, 2016 Feb. 1, 2018. Published August 2016 March 2018. Originally approved in 1997. Last previous edition approved in 2013 as F1473 – 16. DOI: 10.1520/F1473-16.10.1520/F1473-18.

² Lu, X., and Brown, N., "A Test for Slow Crack Growth Failure in Polyethylene Under a Constant Load," Journal of Polymer Testing, Vol 11, pp. 309–319, 1992.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

3.3.1 slow crack growth—the slow extension of the crack with time.

4. Summary of Test Method

4.1 Specimens are prepared from compression molded plaques, precisely notched and then exposed to a constant tensile stress at elevated temperatures in air. Time on test is recorded for tests conducted against a minimum time before failure requirement or the time to complete failure is recorded.

Note 2-Minimum time before failure requirements are found in material or product specifications, codes, etc.

5. Significance and Use

- 5.1 This test method is useful to measure the slow crack growth resistance of molded plaques of polyethylene materials at accelerated conditions such as 80°C, 2.4-MPa stress, and with a sharp notch.
- 5.2 The testing time or time to failure depends on the following test parameters: temperature; stress; notch depth; and specimen geometry. Increasing temperature, stress, and notch depth decrease the time to failure. Material parameters, not controlled by the laboratory, that could impact the test results (time to failure) are: pigment (color or carbon black) and the carrier resin for the pigment, or both. Thus, in reporting the test time or time to failure, all the conditions of the test shall be specified.

Note 3—Time to failure can also be affected by the degree of pigment (color or carbon black) dispersion and distribution within the test specimen. Test Method D5596 and ISO 18553 provide methods for assessing the degree of dispersion and distribution of the pigment

6. Apparatus

- 6.1 Lever Loading Machine, with a lever arm ratio of about 5:1. The tensile load may also be applied directly using dead weights or any other method for producing a constant load. The pull rods on the grips shall have universal action to prevent bending. The grips shall be serrated to prevent slippage. The load on the specimen shall be accurate to at least $\pm 0.5 \%$.
 - 6.2 Furnace, heated by ordinary incandescent light bulbs covered with aluminum foil or any other suitable heating element.
 - 6.3 Temperature Controller, shall be able to control the temperature within ±0.5°C with respect to the set point.
- 6.4 *Temperature-Measuring Device*, a thermometer or a thermocouple which can measure the temperature with an accuracy better than 0.5°C.
 - 6.5 Timer, shall have an accuracy of at least 1 % and shall automatically stop when the specimen fails.
- 6.6 Alignment Jig, as shown in Fig. 1, which aligns the grips and the specimen when the specimen is being tightened in the grips. Alignment jigs which produce the same function may be used.
- 6.7 Notching Machine, for notching the specimen is shown in Fig. 2 or other machines which produce the same results may be used. The notching machine presses a razor blade into the specimen at a speed less than 0.25 mm/min. The depth of the notch is controlled within ± 0.01 mm. The machine is designed so that the main notch and the side notches will be coplanar and the plane of the notching is perpendicular to the tensile axis of the specimen. The thickness of the razor blade is approximately 0.2 mm.

7. Precautions

7.1 The load shall be carefully added to avoid shocking the specimen. When the specimen is inserted in the grips, bending and twisting shall be avoided in order to prevent the premature activation of the notch. Avoid exposure to fluids such as detergents.

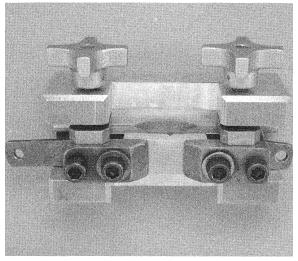
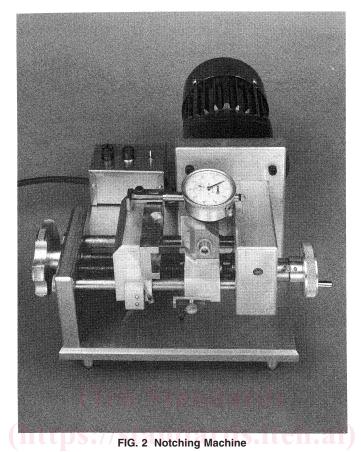


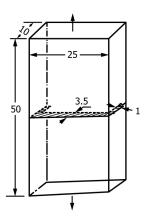
FIG. 1 Alignment Jig



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8. Test Specimens

- 8.1 Specimens are machined from a compression molded plaque of the polyethylene material.
- 8.2 Specimen Geometry—A representative geometry for compression molded plaque specimens is shown in Fig. 3.
- 8.3 Dimensional Requirements:
- 8.3.1 The side groove shall be 1.0 \pm 0.10 mm for all plaque thicknesses.



Legend:
Arrows designate direction of tensile stress.

All dimensions are in millimetres.

t =thickness.

FIG. 3 Representative Geometry for Compression-Molded Specimen

- 8.3.2 The overall length is not critical except that the distance between the notch and the end of a grip should be more than 10 mm. Thicker specimens should have a greater overall length so that the gripped area will be greater in order to avoid slippage in the grip.
 - 8.4 Preparation of Compression Molded Plagues—
- 8.4.1 Polyethylene resins shall be evaluated by using specimens that are machined from compression molded plaques using Practice D4703, except for the following procedures. After the resin is heated to 140 to 160°C, apply and remove the pressure three times. Increase the temperature to 170 to 190°C for 10 to 15 min without pressure. Then apply and remove the pressure three times. The specific temperatures that are used depend on the melt index of the resin, that is, a higher temperature for a lower melt index. The purpose of applying and removing the pressure is to eliminate voids. Turn off the heat and apply pressure. The time to cool between 130 and 90°C shall be greater than 80 min. Alternatively, the time to cool from the molding temperature to about room temperature shall be greater than 5 h. During cooling the pressure is allowed to decrease naturally.
- 8.5 Specimen Notching—The specimen has two types of notches, the main notch and two side notches. The side notches are usually referred to as "side grooves." The depth requirements for these notches are given in Table 1. The main notch is produced by pressing a razor blade into the specimen at a speed of less than 0.25 mm/min. A fresh razor blade shall not be used for more than three specimens and shall be used within one day. The rate of notching for the side grooves is not important. It is important to make the side grooves coplanar with the main notch. Specimens shall be notched at room temperature.

9. Conditioning

9.1 Unless otherwise specified, hold the test specimens for at least 1 h at the test temperature prior to loading. The length of time between notching and testing is not important.

10. Procedure

- 10.1 Calculation of Test Load:
- 10.1.1 Calculate the test load, P, as follows:

 $P = \sigma \times w \times t \tag{1}$

where:

 σ = stress,

w = specimen width, and

t =specimen thickness.

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The variables w and t are based on the specimens prior to notching.

- 10.1.2 If σ has the units of megapascals and w and t are in millimetres, and A is in square millimetres, then P has the units of Newtons. To convert Newtons to pounds, multiply by 0.225. If a lever-loaded machine is used, divide P by the lever arm ratio. The load on the specimen shall be $\pm 0.5\%$ of the calculated load.
- 10.2 *Gripping the Specimen*—Using an alignment jig (Fig. 1), center the specimen in the grips so that the axis of the specimen is aligned with the grips. When the grips are tightened, it is important not to activate the notch by bending or twisting the specimen. The ends of the grips shall be at least 10 mm from the notch.

TABLE 1 Notch Depth as a Function of Specimen Thickness^A

This table is based on the stress intensity being the same for all thicknesses. Thickness, mm Notch Depth, mm 4 00 1.90 5.00 2.28 6.00 2.50 7.00 2 80 8.00 3.09 9.00 3.30 10.00 3.50 3.70 11.00 12.00 3.90 13.00 4.18 14.00 4 39 4.48 15.00 16.00 4 65 17.00 4.88 18.00 4.95 19.00 5.09 20.00 5.20

^A For an intermediate thickness, linearly interpolate to obtain the notch depth. The notch depth in the specimen shall be within± 0.05 mm of the interpolated value.

- 10.3 Loading the Specimen—When the specimen in the grips is removed from the alignment jig and transferred to the testing machine, take care that the notch is not activated by bending the specimen. Apply the load after the specimen has been held for at least 1 h at the test temperature. Apply the load gradually within a period of about 5 to 10 s without any impact on the specimen.
- 10.4 *Temperature Measurement*—Place the thermocouple or thermometer near the notched part of the specimen. Periodically record the temperature with a frequency that depends on the length of the test.
- 10.5 When testing is stopped before failure or when the specimen fails, record the time on test, or the time to failure. Failure occurs when the two halves of the specimen separate completely or extensive deformation occurs in the remaining ligament.

11. Report

- 11.1 Compression-molded test specimens shall be identified by the polyethylene material source (resin manufacturer or other source) and lot number. lot number as well as information of the color (for example, natural, black, blue etc.) of the specimen being tested.
 - 11.2 Stress based on the unnotched area.
 - 11.3 Depth of main notch and side grooves.
 - 11.4 Calculated load and cross-sectional dimensions of the specimen.
 - 11.5 Test temperature.
 - 11.6 Time on test or time to failure.
 - 11.7 Date and time for the beginning and ending of the test.

12. Precision and Bias

- 12.1 *Precision*—A round robin was conducted with seven laboratories and used three resins from different producers. The standard deviation of the average values within laboratories is ± 16 %. The standard deviation of the average values between laboratories is ± 26 %.
- 12.2 *Bias*—No statement on bias can be made because there is no established reference value. The test method originated at the University of Pennsylvania. If the test results from about eight years of testing at the University of Pennsylvania can be used as reference values, then there is no bias in the results from the different laboratories with respect to the results at the University of Pennsylvania. If the test results from the University of Pennsylvania can be used as a reference, then there is no bias for the round robin starting with pellets.⁵

13. Keywords

13.1 fracture; notch testing; pipes; polyethylene; resin; slow crack growth

ANNEX

(Mandatory Information)

A1. MEASUREMENT OF SPECIMEN THICKNESS DIMENSION

A1.1 Scope

A1.1.1 The following procedures for measurement shall be used when required by the standard for the product being tested. These requirements are in addition to those in the main body of this test method.

A1.2 Referenced Documents

A1.2.1 ASTM Standards:²

D618 Practice for Conditioning Plastics for Testing

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F17-1043. This report is based on a round robin of seven laboratories starting with pellets.

A1.3 Conditioning

A1.3.1 Conditioning Temperature and Humidity—Condition the test specimens in accordance with Practice D618 Procedure A without regard for humidity unless otherwise specified by contract or when required by the standard for the product being tested.

A1.4 Apparatus

A1.4.1 The following apparatus shall be used—Flat-Anvil Micrometer with an accuracy of at least \pm 0.001 in. (\pm 0.025 mm).

A1.5 Dimensioning

- A1.5.1 Measure the thickness of each specimen prior to notching to the nearest 0.001 in. (0.025 mm) at the center of each specimen.
- A1.5.2 If the thickness dimension has a required value in the standard for the product being tested, then report the thickness to the same decimal place required by that standard.
- A1.5.3 If the thickness dimension has a required precision in the standard for the product being tested, then report the precision of the apparatus used for measurement.

APPENDIX

(Nonmandatory Information)

X1. TESTING SPECIMENS FROM PIPE

- X1.1 Scope—Test Method F1473 has been used to measure the slow crack growth resistance of specimens from pipe.
- X1.1.1 Test results are affected by size, specimen geometry, molecular orientation, and other processing effects.
- X1.1.2 Extrusion generally aligns polyethylene molecules parallel to the extrusion direction. Notching perpendicular to the extrusion direction (Fig. X1.1(a)) generally gives higher results than notching parallel to the extrusion direction (Fig. X1.1(b)).
- X1.1.3 Values obtained from tests of specimens cut from pipe can vary significantly from values obtained from tests of specimens machined from a compression molded plaque of the resin.
- X1.2 Significance and Use—Test results may be useful for research, or for comparison or evaluation of resin or processing effects on slow crack growth resistance.
- X1.2.1 While the resin is the primary factor in slow crack growth resistance, when tests are conducted on specimens from pipe, pipe size, pipe wall thickness, extrusion equipment, and processing can affect test results. These influences can be addressed by consistency and uniformity in preparing, loading, and notching specimens. This is especially important when testing is for the purpose of evaluation or comparison.
- Note X1.1—Many combinations of different types of extrusion equipment, tooling, and processing conditions are used to extrude polyethylene pipe. Differences in extrusion equipment, tooling, and processing conditions are known to affect the results when specimens cut from pipe are tested in accordance with this test method.

X1.3 Specimen Preparation:

X1.3.1 When a section of the pipe wall is to be tested, cut sections or strips from the pipe. Sections or strips should be cut 4 to 6 mm wider than the required specimen width, then deburred, and machined to the specimen width.