

# Designation: F2136 - 08 (Reapproved 2015)

# Standard Test Method for Notched, Constant Ligament-Stress (NCLS) Test to Determine Slow-Crack-Growth Resistance of HDPE Resins or HDPE Corrugated Pipe<sup>1</sup>

This standard is issued under the fixed designation F2136; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

# 1. Scope

- 1.1 This test method is used to determine the susceptibility of high-density polyethylene (HDPE) resins or corrugated pipe to slow-crack-growth under a constant ligament-stress in an accelerating environment. This test method is intended to apply only to HDPE of a limited melt index and density range as defined in AASHTO Standard Specification M 294. This test method may be applicable for other materials, but data are not available for other materials at this time.
- 1.2 This test method measures the failure time associated with a given test specimen at a constant, specified, ligament-stress level.
- 1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.
- 1.4 Definitions are in accordance with Terminology AASHTO Standard Specification M 294, and abbreviations are in accordance with Terminology D1600, unless otherwise specified.
- 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 and health practices and determine the applicability of regulatory limitations prior to use.

## 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D1600 Terminology for Abbreviated Terms Relating to Plastics

D1822 Test Method for Tensile-Impact Energy to Break Plastics and Electrical Insulating Materials

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

D5397 Test Method for Evaluation of Stress Crack Resistance of Polyolefin Geomembranes Using Notched Constant Tensile Load Test

E4 Practices for Force Verification of Testing Machines
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
F412 Terminology Relating to Plastic Piping Systems
2.2 Other Document:

AASHTO Standard Specification M 294<sup>3</sup>

## 3. Summary of Test Method

3.1 This test method subjects a dumbbell-shaped, notched test-specimen (Fig. 1) to a constant ligament-stress in the presence of a surface-active agent at an elevated temperature. It differs from Test Method D5397 in that a constant ligament stress is used instead of a constant tensile load.

## 4. Significance and Use

- 4.1 This test method does not purport to interpret the data generated.
- 4.2 This test method is intended to compare slow-crack-growth (SCG) resistance for a limited set of HDPE resins.
- 4.3 This test method may be used on virgin HDPE resin compression-molded into a plaque or on extruded HDPE corrugated pipe that is chopped and compression-molded into a plaque (see 7.1.1 for details).

## 5. Apparatus

5.1 Blanking Die—A die suitable for cutting test specimens. Acceptable dies are: the type L die per Test Method D1822, with holes drilled or punched in the tab areas after die cutting; a die with the dimensions and tolerances specified in Fig. 2.

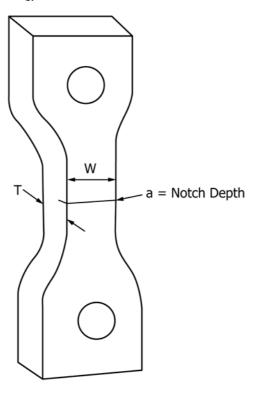
<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee F17 on Plastic Piping Systems and is the direct responsibility of Subcommittee F17.40 on Test Methods.

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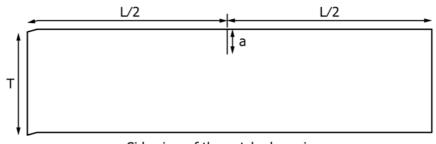
<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>3</sup> Available from American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001, http://www.transportation.org.





Front view of the notched specimen  $A = (T-a) \times W$ 



Side view of the notched specimen

T = thickness.
W = specimen width.

Note 1—The test specimen is intended to have the same geometry used for Test Method D5397 specimens. The length of the specimen can be changed to suit the design of the test apparatus. However, there should be a constant neck section with length at least 0.5 in. (13 mm) long.

Note 2—It is preferable to modify the specimen die so that the attachment holes are punched out at the same time as the specimen rather than punching or machining them into the specimen at a later time. If the attachment holes are introduced at a later time, it is extremely important that they be carefully aligned so as to avoid adding a twisting component to the stress being placed on the specimen.

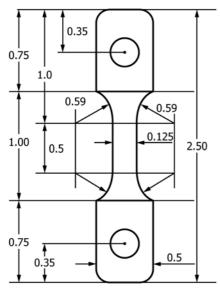
FIG. 1 Notching Position

- 5.2 Stress-Crack Testing Apparatus—A lever loading machine, with a lever arm ratio of 2:1 to 5:1 similar to that described in Test Method D5397. Alternatively, the tensile load may be applied directly using dead weights or any other method for producing a constant ligament stress. Determine the zero-load offset and lever-arm ratio for each test station, using a force standard that complies with Practices E4. The load on the specimen shall be accurate to 0.5 % of the calculated or applied load. The bath solution temperature shall be set at 122  $\pm$  2°F (50  $\pm$  1°C).
- 5.3 *Notching Device*—Notch depth is an important variable that must be controlled. Paragraph 7.2.1 describes the notching

procedure and type of apparatus used. The approximate thickness of the blade should be 0.2 to 0.3 mm.

Note 1—A round robin was conducted to determine the effect of types of blades on the notch depth. In this study, several types of steel blades (single-edge, double-edge, and so forth) from various manufacturers were used by the round-robin participants. The round robin consisted of seven laboratories using two types of resins molded into plaques. The standard deviation of the test results within laboratories is less than  $\pm 10~\%$ .

- 5.4 *Micrometer*, capable of measuring to  $\pm 0.001$  in.  $(\pm 0.025 \text{ mm})$ .
- 5.5 *Microscope*, equipped with micrometer or an equivalent device capable of accurately measuring the notch depth.



Note 1—Dimensions are in inches with tolerance of  $\pm 0.005$  in., except specimen width, which has a tolerance of  $\pm 0.001$  in.

FIG. 2 Specimen Geometry—Test Specimen Dimensions

- 5.6 Compression-Molding Press and Suitable Chase for Compression-Molding the Specimens, in accordance with Practice D4703.
  - 5.7 Metal Shot, for weight tubes.
- 5.8 *Electronic Scale*, for measuring shot weight tubes capable of measuring to  $\pm 0.1$  g.
- 5.9 *Timing Device*, capable of recording failure time to the nearest 0.1 h.

### 6. Reagents

6.1 The stress-cracking reagent shall consist of 10% non-ylphenoxy poly (ethyleneoxy) ethanol by volume in 90% deionized water. The solution level is to be checked daily and deionized water used to keep the bath at a constant level.

#### 7. Procedure

- 7.1 Specimen Preparation:
- 7.1.1 Compression-mold pellet specimens (virgin resin) or chopped pipe into 0.075-in. (1.9-mm) sheet in accordance with Procedure C of Practice D4703, except that the pellets do not have to be roll-milled prior to being compression-molded. The rate of cooling shall be 27 +/- 3.6°F (15  $\pm$  2°C) per minute. If desired, the sheet may be trimmed by 0.6 in. (15 mm) on each side in order to avoid any edge effects. Since pipes have extrusion-induced orientation that can significantly affect the test results, it is necessary to remove the orientation effect by molding into a plaque. Chop and mold a pipe specimen in accordance with the following procedure. Cut 1-in. (25-mm) wide sections from the pipe along its longitudinal axis. To randomize the orientation, cut these sections into smaller pieces until there is about 1 lb (0.5 kg) of material. These sections represent a complete cross-sectional sample from the inside to the outside of the pipe specimen. Compression mold

a plaque as previously stated. If different materials are used for the inner and outer wall of dual wall pipe, each wall must be tested separately.

7.1.2 Die cut test specimens from the sheet, and make holes in the specimen as shown in Fig. 1.

7.1.3 Specimen tolerances are as follows:

Length =  $2.36 \pm 0.01$  in.  $(60.00 \pm 0.25$  mm) Width =  $0.125 \pm 0.001$  in.  $(3.20 \pm 0.02$  mm) Thickness =  $0.075 \pm 0.003$  in.  $(1.90 \pm 0.08$  mm)

# 7.2 Notching:

7.2.1 Notch specimens across the center of the 0.125-in. (3.20-mm) wide, 0.500-in. (12.7-mm) long reduced section as shown in Figs. 1 and 2. Cut the notch perpendicular to the plane defined by specimen length and width, and align at a right angle to the direction of load application. Cut the notch at a maximum rate of 0.1 in./min (2.5 mm/min) to a depth of

$$a = 0.20 \times T \tag{1}$$

where:

a =notch depth, and

T = measured thickness of the specimen.

Control notch depth to  $\pm 0.001$  in. ( $\pm 0.025$  mm) by measuring the notch depth with a microscope.

7.2.2 No single razor blade shall be used for more than ten test specimens.

- 7.3 Calculation of Test Load:
- 7.3.1 For each specimen, measure the reduced section width (W), thickness (T), and notch depth (a) to the nearest 0.001 in. (0.025 mm) using a micrometer and a microscope, or determine the width (W) with a micrometer and determine the ligament thickness directly with a microscope to the nearest 0.0001 in. In the latter case, substitute the ligament thickness in inches for the term (T-a) in Eq 2.

7.3.2 At each loading point, determine the weight that must be hung on the lever arm to produce the required ligament-stress directly, by installing a calibrated load cell in the position of the future test specimen and preparing the necessary weight accurately enough that the ligament stress does not vary by more than  $\pm 0.5$  %. The appropriate load cell reading is as follows:

Required load cell reading lbs (grams) = 
$$(T - a) W S$$
 (2)

and

P = the necessary weight to be applied to the lever at the loading station to produce the required load cell reading as measured directly by the load cell.

where:

*P* is measured directly by adding weight, as necessary at each loading station while the load cell is in place,

W =cross-sectional width of the test specimen,

a =the depth of the notch measured in accordance with 7.3.1.

T = the thickness of the test specimen, and

S = specified ligament stress, psi (MPa).

Each test weight so determined is to be labeled (or otherwise correlated to each test position) and applied to the appropriate lever arm on the test apparatus. Note 2--S = the specified ligament-stress. It is the stress at the notch location within each test specimen during the test. It may be expressed as a percent (%) of the reference yield stress of 4000 psi (27.5 MPa). The specified ligament stress is selected at a level that is high enough to provide a differentiation between materials that provide acceptable stress-crack resistance and those that do not, within a reasonable testing time period. The reference yield stress of 4000 psi has been selected for all resins meeting AASHTO M 294 density specifications of 0.945-0.955 g/cc. This value is near the actual yield stress levels of PE materials representing the upper end of this density range.

- 7.4 NCLS Testing:
- 7.4.1 Maintain temperature in the bath at  $122 \pm 2^{\circ}F$  (50  $\pm 1^{\circ}C$ ).
  - 7.4.2 Test five specimens at a single ligament stress level.
- 7.4.3 Determine the weight to be placed on each specimen, and load the weight tubes with shot. Do not attach the shot tube to the lever arm.
- 7.4.4 Attach the specimens to the loading frame. Take care that the notch is not activated by bending the specimen. Lower the specimen into the bath, and condition the specimens in the bath for at least 30 min.
  - 7.4.5 Reset the specimen timer to zero.
- 7.4.6 Check that the weight is the correct weight for the particular specimen, and carefully connect the weight tube to the appropriate lever arm for the specimen. Apply the load gradually within a period of 5 to 10 s without any impact on the specimen.
  - 7.4.7 Start the specimen timer immediately after loading.
- 7.4.8 Record the time to failure of each specimen to the nearest 0.1 h.

## 8. Report

- 8.1 Report the following information:
- 8.1.1 All details necessary for complete identification of the material tested (density, melt index, lot number, and so forth).
  - 8.1.2 Reference to this ASTM Test Method (F2136).
- 8.1.3 The load placed on each level in accordance with Equation and cross-sectional dimension of each specimen.

- 8.1.4 The ligament-stress (in MPa or psi) based on the cross-sectional area of the test specimen.
  - 8.1.5 Test temperature.
- 8.1.6 If applicable, the extrusion or molding from which the test pieces has been taken.
- 8.1.7 The failure time for each of the five specimens and the arithmetic average of each specimen set of five specimens. The arithmetic average shall be reported as the NCLS value of the resin or pipe under test.

### 9. Precision and Bias<sup>4</sup>

9.1 *Precision*—Based on Practice E691, a nine-laboratory round-robin conducted on four HDPE materials, the precision (one standard deviation) of this test method is summarized as follows. This precision was determined using the Practice E691 "Interlaboratory Data Analysis Software" computer program. The within-laboratory repeatability standard deviation (Sr) and between-laboratory reproducibility standard deviation (SR) are based on reporting the average of five specimens as one data set.

HDPE Material	Repeatability, (Sr), Within laboratory, %	Reproducibility, (SR), Between laboratory, %
Α	20	50
В	24	39
С	11	45
D	6	27

9.2 *Bias*—Data obtained using this test method are believed to be reliable since accepted techniques of analysis are used. Since no referee method is available, no bias statement can be made.

# 10. Keywords

10.1 constant ligament-stress; corrugated HDPE pipe; slow-crack-growth resistance

# APPENDIX

(Nonmandatory Information)

### X1. Example of Load Calculation

### X1.1 Calculate load as follows:

Load (grams) = 
$$\frac{S*(T-a)*W}{\left[(MA)*(9.81)\right]} \times 1\ 000 - \frac{CF}{MA} \text{ (SI units)}$$
(X1.1)

or

Load (lb) = 
$$\frac{S*(T-a)*W - CF}{(MA)}$$
 (Inch – pound units) (X1.2)

#### where:

a =notch depth, in. (mm),

MA = mechanical advantage of the apparatus (equipment dependent),

W = specimen width, in. (mm),

T =specimen thickness, in. (mm),

S = constant ligament-stress, psi (MPa), and

*CF* = correction factor for the arm weight.

<sup>&</sup>lt;sup>4</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F17-1046.

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