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An American National Standard

Standard Test Method for Evaluating the Oxidative Resistance of Polyethylene (PE) Pipe to Chlorinated Water¹

This standard is issued under the fixed designation F2263; 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-Scope*

1.1 This test method describes the general requirements for evaluating the long-term, chlorinated water, oxidative resistance of polyethylene (PE), used in cold water supply or service systems by exposure to chlorinated water. This test method outlines the requirements of a pressurized flow-through test system, typical test pressures, test-fluid characteristics, failure type, and data analysis.

Note 1—Other known disinfecting systems (chlorine dioxide, ozone, and chloramine) are currently used for protection of potable water; however, free-chlorine is by far the most common system in use today. Disinfecting systems other than chlorine have not been evaluated by this method.

1.2 Guidelines and requirements for test temperatures, test hoop stresses, and other test criteria have been established by prior testing of PE pipe. Other related system components that typically appear in a PE cold water supply or service system can be evaluated with the PE pipe. When testing PE pipe and fittings as a system, it is recommended that the anticipated end-use fitting type(s) and material(s) be included in the test circuit since it is known that some fitting types and materials can impact failure times. Specimens used shall be representative of the piping product(s) and material(s) under investigation.

Note 2—The procedures described in this test method (with some modifications of test temperatures or stresses, or both) have been used to evaluate pipes manufactured from polybutylene (PB), crosslinked polyethylene (PEX), polypropylene (PP), multilayer (polymer-metal composite), copper, and stainless steel

- 1.3 This test method is applicable to PE pipe and systems used for transport of potable water containing free-chlorine for disinfecting purposes. The oxidizing potential of the test-fluid specified in this test method exceeds that typically found in potable water systems across the United States.
- 1.4 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.
- 1.5 The following precautionary caveat pertains only to the test method portion, Section 12, of this specification. 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:²

D1600 Terminology for Abbreviated Terms Relating to Plastics

D2122 Test Method for Determining Dimensions of Thermoplastic Pipe and Fittings

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

F412 Terminology Relating to Plastic Piping Systems

2.2 ISO Standards:

ISO 9080 Thermoplastic Pipe for Transport of Fluids—Methods of Extrapolation of Hydrostatic Stress Rupture Data to Determine the Long Term Strength of Thermoplastic Pipe³

2.3 Plastics Pipe Institute (PPI) Document:

TN-16 Rate Process Method for Projecting Performance of Polyethylene Piping Components⁴

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 Nov. 1, 2014 Published December 2015 Published December 2015 Published December 2015 Published December 2016 Published Publis

² 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.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

⁴ Available from Plastics Pipe Institute (PPI), 105 Decker Court, Suite 825, Irving, TX 75062, http://www.plasticpipe.org.

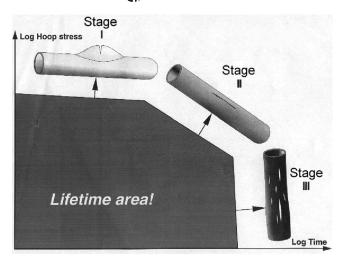


FIG. 1 Pictorial Illustration of Failure Types

2.4 American Water Works Association (AWWA) Document: 1996 WATER: STATS Survey⁵

3. Terminology

- 3.1 Definitions:
- 3.1.1 Definitions are in accordance with Terminology F412 and abbreviations are in accordance with Terminology D1600, unless otherwise indicated.
- 3.1.2 brittle failure (Stage II), n—failure in the pipe wall that is characterized by little or no material deformation in the failure area and is the result of a single crack emanating from the interior of the pipe to the outside surface typically resulting in a pinhole leak, see Fig. 1.
- 3.1.2.1 oxidatively induced brittle failure (Stage II), n—a type of brittle failure (Stage II) that is characterized by embrittlement of the interior surface of the pipe. This type of Stage II failure is developed through oxidative degradation of the surface of the inner wall of the piping material. Guidelines for identifying the failures are provided in Appendix X3.
- 3.1.3 *cold water supply or service system, n*—a combination of components such as pipe, fittings, valves, and so forth, that when installed as a complete system, make up the water supply system. 263–14
- 3.1.4 *ductile failure (Stage I)*, *n*—failure in the pipe wall that is characterized by obvious localized deformation of the material visible with the unaided eye, see Fig. 1. Ductile failures produced with this test method shall not be used for data analysis.
- 3.1.5 environmental or oxidative failure (Stage III), n—failure in the pipe wall characterized by a large number of cracks emanating from the interior surface of the pipe wall, see Fig. 1.
- 3.1.6 *long-term oxidative resistance, n*—the extrapolated time-to-failure prediction as determined by analysis of time-to-failure test data by multiple linear regression utilizing the rate process method of PPI TN-16 or three parameter model of ISO 9080.
- 3.1.7 *multiple linear regression*, *n*—a three or four coefficient mathematical model used to analyze time-to-failure data from different temperatures and stresses to extrapolate projected time-to-failure at selected temperatures or stresses.
- 3.1.8 oxidation reduction potential (ORP), n—oxidation reduction potential (ORP), n-a measure of the total oxidizing power of a solution by means of a platinum-redox electrode. For a further explanation of ORP see Appendix X2.
 - 3.1.9 unaided eye, n—observable without visual enhancement beyond correction for normal vision.

4. Summary of Test Method

4.1 The PE pipe/fitting assemblies are exposed to pressurized test-fluid until failure. All time-to-failure data used for analysis shall be the result of the same failure mode, either all stage oxidatively induced Stage II or all stageStage III. A minimum number of test temperature and hoop stress conditions are required to allow accurate data analysis and time-to-failure extrapolations. If using only stage II failure data, no lifetime extrapolations can be made without further validation.

5. Significance and Use

5.1 Environment or oxidative time-to-fail data derived from this test method, analyzed in accordance with Section 13, are suitable for extrapolation to typical end-use temperatures and hoop stresses. The extrapolated value(s) provides a relative indication

⁵ Available from American Water Works Association (AWWA), 6666 W. Quincy Ave., Denver, CO 80235, http://www.awwa.org.

of the resistance of the tested PE pipe or system to the oxidative effects of chlorinated water for conditions equivalent to those conditions under which the test data were obtained. The performance of a material or piping product under actual conditions of installation and use is dependent upon a number of factors including installation methods, use patterns, water quality, nature and magnitude of localized stresses, and other variables of an actual, operating cold water supply or service system that are not addressed in this test method. As such, the extrapolated values do not constitute a representation that a PE pipe or system with a given extrapolated time-to-failure value will perform for that period of time under actual use conditions.

5.2 This test method has been generally used for evaluating oxidative (stage III) failure data. For some systems being tested accelerated stage II failures can occur. These failures can also be analyzed using the regression analysis outlined in Section oxidatively induced Stage II or Stage III failure data. 13. Extrapolation of this data may need to be validated prior to making any lifetime predictions.

6. Apparatus

- 6.1 Pressurized Flow-Through Test System—A system comprised of the necessary pump(s), fittings, piping, heaters, sensors, and meters that is capable of maintaining the required test pressures within the tolerance specified in 9.1.3, the required test temperatures within the tolerance of 9.1.2, and flow the test-fluid through the specimens continually at a flow rate within the tolerance specified in 9.1.4. Cyclic pressure variations, such as those produced by some pumping systems, shall not produce pressure excursions that exceed the tolerance stated in 9.1.3.
- 6.2 *Specimen Holders*—Test specimens shall be supported to minimize or eliminate externally induced stresses. Specimens shall be allowed to freely expand bi-directionally.

7. Sampling, Test Specimens, and Test Units

- 7.1 Sampling—Select at random, a sufficient amount of pipe to satisfy the specimen requirements of this test method. When testing as a system, randomly select a sufficient quantity of fittings.
- 7.2 Test Specimen Size—The recommended minimum pipe size is ½ CTS. CTS, and common test sizes are ½ in. CTS and 4 in. IPS. The PE pipe specimens shall be 12 to 18 in. (300 to 460 mm) in length between fitting closures or between fitting joints.
- 7.2.1 Dimensions Measurement—Measure and record the critical dimensions for pipe and fittings. For pipe, measure the average outside diameter and wall-thickness in accordance with Test Method D2122. For fittings, measure those dimensions critical to the function of the joint, as well as minimum body wall thickness.
- 7.3 Testing as a System—When testing PE pipe and related system components (such as fittings) as a system, the other components shall be attached to the PE pipe in the same manner as in actual service. For fittings, the particular fitting style shall be installed in accordance with the manufacturer's instructions or the ASTM specification when applicable.
- 7.4 Minimum Required Test Units—A minimum of six test units is required. A test unit is comprised of two or more individual time-to-failure data points at the same temperature and hoop stress condition. Obtaining additional data points at each temperature/hoop stress condition will benefit statistical reliability of the analysis of the resultant data.
- 7.4.1 *Test Unit Distribution*—Time-to-failure data points shall be obtained at 2 test hoop stresses at each of a minimum of 3 test temperatures for a minimum of 12 data points. As an alternate, obtain time-to-failure data for the temperature/hoop stress combinations of the three-temperature matrix of PPI TN-16, see Note 3. Hoop stresses shall be separated by a least 80 psi (0.55 MPa).
 - Note 3—When using the PPI TN-16 matrix, Temperature T_3 , which requires testing at only one stress, refers to the lowest test temperature.
- 7.4.2 Test Temperature Selection —Temperatures of 90°C (194°F), 80°C (176°F), and 70°C (158°F) have been utilized in prior testing of PE, see Note 4. Adjacent test temperatures shall be separated by at least 18°F (10°C). Other test temperatures may be used, but the maximum test temperature shall not exceed 95°C (203°F).
- Note 4—Prior testing indicates that for the test temperatures stated in 7.4.2, hoop stresses to yield oxidatively induced Stage II or III-failures within reasonable testing times are between 120360 psi (830 kPa)(2.48 MPa) and 480 psi (3.31 MPa). For a true-SDR 9 tube, those hoop stresses correspond to test pressures of 3090 psig (207(620 kPa) to 120 psig (830 kPa). Target lower stresses to yield Stage III failures. If a selected test hoop stress produces Stage I or Stage II failures, the stress will need to be reduced to produce all oxidatively induced Stage II or all Stage III failures at all temperatures.
- 7.4.2.1 *Relationship of Internal Pressure to Hoop Stress*—The hoop stress in the pipe wall is calculated by the following expression, commonly known as the ISO equation:

$$2S/P = DR - 1 \tag{1}$$

or

$$2S/P = (D_o/t) - 1 \tag{2}$$

where:

S = stress in the circumferential or hoop direction, psi (MPa),

P = internal pressure, psig (kPa),



t = minimum wall thickness, in. (mm),

DR = dimension ratio, DR, and

 D_o = average outside diameter, in. (mm).

8. Calibration and Standardization

8.1 *Measuring Equipment*—All measuring and testing equipment having an effect on the accuracy or validity of the calibrations or tests shall be calibrated or verified, or both, before being put into service.

9. Test Fluid

- 9.1 Internal Test Fluid—The test fluid shall be reverse osmosis (RO) or deionized (DI) water prepared in accordance with 9.1.1.
- 9.1.1 RO or DI Water Test-Fluid Preparation—Test fluid prepared from RO or DI water shall have a pH in the range from 6.5 to 8.0 and contain 2.5 ppm to 5 ppm (milligrams per litre) of free-chlorine. Testing shall be conducted with the same nominal pH and free-chlorine concentration for all test units. The chosen pH shall be maintained to \pm 0.2 and the chosen free-chlorine concentration shall be maintained to \pm 0.2 ppm. The pH and free-chlorine concentration combination shall yield a minimum ORP of 825 mV for the test fluid.
- 9.1.2 Test Fluid Temperature Control—The test fluid entering each specimen shall be maintained to ± 1.8 °F (± 1 °C) of the test temperature.
 - 9.1.3 Pressure Control—The pressure of the test fluid shall be maintained to ± 3 psig (± 20.69 kPa).
- 9.1.4 Test Fluid Flow Rate—The flow rate shall be selected such that the average ORP of the test fluid shall yield a minimum velocity of 0.12 fps (0.04 mps). For the nominal sizeexiting the test specimens ½ in., SDR 9 tubing, this corresponds to a flow rate of 0.06 gpm (0.23 LPM). The formula used to calculate the flow rates for other sizes and DRs is as follows: remains above 825 mV. The same nominal flow rate shall be used for all test specimens. Flow rate shall be

$$\frac{\pi (id/2)^{2*}FPS*720}{231} = \text{gpm} \tag{3}$$

maintained \pm 10% of the nominal flow rate.

where

id = measured inside diameter of the tubing, in.

Note 5—It has been established that, for nominal size ½ in. CTS SDR 9 tubing, a minimum flow rate of 0.06 usgpm (0.23 LPM), and for 4 in. IPS pipe, a minimum flow rate of 0.125 usgpm (0.47 LPM), meets this requirement.

Note 6—The test flow rates are not intended to replicate application in-service flow rates. A continuous flow of test fluid is required to replenish the chlorine consumed in the test. The flow rate is selected to maintain the oxidative strength (ORP) of the fluid within specification. Different test configurations may require different flow rates to achieve this objective. The intent of 9.1.4 is to verify the chosen flow rate meets the ORP requirement at the highest temperature and then use the same flow rate for all tests. Re-verification would be conducted with significant changes in the test configuration.

- 9.2 Test Fluid Instrument Accuracy:
- 9.2.1 pH—The pH measurement and control instruments shall have an accuracy of 0.1 pH or better.
- 9.2.2 Free-Chlorine—Free-chlorine content measurement and control instruments shall have an accuracy of 0.1 ppm or better.
- 9.2.3 *ORP*—The ORP measurement and control instruments shall have an accuracy of ± 10 mV or better.

10. External Environment

10.1 The exterior environment shall be air and shall be maintained at the target temperature of the test fluid temperature ± 4.5 °F (± 2.5 °C). Direct, forced-air heating of the specimens shall not be used.

11. Specimen Positioning

11.1 The specimens can be positioned vertically or horizontally. Horizontal positioning requires special attention to insure that all entrapped air has been removed prior to starting the test. For vertically positioned specimens, the test fluid shall flow into the specimens from the lower end.

12. Procedure

- 12.1 Perform the test procedure in accordance with 12.2 12.4 for the test units specified in 7.4 with a test fluid as specified in Section 9.
- 12.2 After connecting the specimens to the flow-through apparatus, purge the specimens of all entrapped gas and start the flow of the test-fluid through the specimens at a temperature or pressure, or both, 40 to 50 % less than the test condition. Over the next 1 to 3 h, gradually increase the temperature and pressure of the test fluid to the test condition. When the test fluid reaches the test condition temperature, pressure, and flow rate, and the external environment has reached the test temperature in accordance with Section 10, register the start time.



- 12.3 Maintain the test condition until all of the specimens have failed. Any loss of fluid through the wall of the pipe or assembly constitutes a failure. Record the time-to-failure for each failed specimen within ± 1 % of the test time for the specimen. When multiple specimens are connected end-to-end, remove each failed specimen and continue the test until all remaining specimens at the conditions have failed.
- 12.4 Record the time in hours and a description for each failure. The description of each failure shall include: linear location from flow inlet, circumferential position, and initiation point (inside or outside of tube). For accurate test life extrapolation, all of the failures must be the same type. Mixed mode failures and failures Failures initiated from the outside of the tube shall not be used for data analysis, see Note 57.

Note 7—Numerous failures occurring predominantly in approximately the same position on the tube circumference should be examined carefully. When there is an indication that the failures are attributable to the design or operation of the test, these values should be discarded unless it can be demonstrated that the testing provided a more conservative estimate of the oxidative resistance.

13. Calculation

- 13.1 Regression Analysis—Perform a multiple linear regression on the time-to-failure data in accordance with the rate process method PPI-TN-16 or three parameter model of ISO 9080. The correlation coefficient (R² value) of the data shall be greater than 0.9 to ensure accuracy of the time-to-failure extrapolations. If it can be demonstrated that the four-coefficient regression equation in three parameter model of ISO 9080 provides a better fit to the data set, then the coefficients, correlation (R² value), and extrapolated times-to-failure from that model shall be used and reported.
- 13.2 *Pressure Calculations*—Using the coefficients from 13.1, calculate the hoop stress (psi) and internal pressure (psig) corresponding to a time of 50 years for the DR of the tested specimens at a temperature of 73°F (23°C). If using only type II failure data, it is not possible to make a lifetime extrapolation without data for the stage III failure of the pipe tested.

14. Report

- 14.1 Report Content—Report the minimum information as required in 14.2 14.9.
- 14.2 Laboratory name and location and starting and ending dates of the test.
- 14.3 Chlorine source (that is, chlorine gas, hypochlorite, and so forth)
- 14.4 Identification of the PE pipe in the report shall include: pipe nominal size and DR or wall thickness specification; average outside diameter and minimum wall thickness of each specimen; pipe manufacturer's name, trade designation and pipe lot number (if applicable); resin manufacturer's name, compound designation, and lot designation (for in-plant compounded materials, resin manufacturer's name may be omitted).
- 14.5 Identification of fitting(s) tested with the PE pipe (if applicable) shall include: manufacturer's name and model or designation, fitting type, material, and ASTM standard designation (if applicable).
- 14.6 All test conditions, including: test fluid temperature/internal pressure combinations; specimen external air temperature; test fluid free-chlorine concentration and pH; test fluid ORP; water type (tap, RO or DI); flow rate; and specimen position (horizontal or vertical). Where applicable, report the minimum, maximum, and average values for each parameter.
 - 14.7 A table of the test temperatures, hoop stresses, and failure times for all specimens tested.
 - 14.8 A description of each failure in accordance with 12.4.
- 14.9 A summary of the regression analysis including: coefficients; correlation coefficient (R^2 value); hoop stress and internal pressure calculation at 50 years and 73°F (23°C).

15. Precision and Bias⁶

- 15.1 The precision of this test method is based on an Interlaboratory study of F2263, Standard Test Method for Evaluating the Oxidative Resistance of Polyethylene (PE) Pipe to Chlorinated Water, conducted in 2005. Each of three laboratories tested one material for chlorine resistance, at one temperature (90°C), and one pressure (60 psig). Each test result reported is an individual analytical determination. Participating laboratories reported 5 or 6 test results for each material, in hours until failure.
- 15.1.1 Repeatability—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.
- 15.1.2 *Reproducibility*—Two test results should be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.
 - 15.1.2.1 Average times to failure for each of the three laboratories were 1045, 1158, and 1373 hours respectively.

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F17-1049.

- 15.1.3 Any judgment in accordance with these two statements would have an approximate 95% probability of being correct.
- 15.2 Bias—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

TABLE 1 Resistance of PE Pipe to Chlorinated Water (in hours)

Material	Average	RepeatabilityRepeatability		ReproducibilityReproducibility	
		Standard	Limit	Standard	Limit
		Deviation		Deviation	
	\bar{X}	Sr	r	SR	R
PE pipe	1192	48.9	137	172	483

15.3 The precision and bias statements were determined through statistical examination of 17 results, from 3 laboratories, on 1 material. Because the number of participating laboratories was limited, these statements do not meet the minimum requirements for determining precision prescribed in Practice E691.

16. Keywords

16.1 chlorine resistance; oxidative resistance; pipe; polyethylene

APPENDIXES

(Nonmandatory Information)

X1. SUMMARY OF FORMULAS

X1.1 Multiple Linear Regression—The three-parameter, rate process equation from PPI TN-16 and ISO 9080 is expressed as follows:

$$Log_{10}ft = A + B/T + C(Log_{10}S)/T$$
(X1.1)

where:

ft = time to failure, h,

T https://st= absolute temperature K° , ndards/sist/cd568770-98d3-4235-a423-cd71c9324328/astm-f2263-14

S = hoop stress, psi (MPa), and

A, B, and C = coefficients derived from a particular data set.

X1.1.1 The four-parameter, multiple linear regression equation from ISO 9080 is expressed as follows:

$$Log_{10}ft = c_1 + c_2/T + c_3 Log_{10}S + c_4(Log_{10}S)/T + e$$
(X1.2)

where:

 $c_1 - c_4$ = coefficients derived from a particular data set, and

e = an error variable, having a Laplace-Gauss distribution with zero mean and constant variance.