



Standard Test Method for Evaluating the Oxidative Resistance of Multilayer Polyolefin Tubing to Hot Chlorinated Water¹

This standard is issued under the fixed designation F 2330; 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 describes the general requirements for evaluating the long-term, chlorinated water, oxidative resistance of multilayer tubing which includes an inner layer of a polyolefin material such as PE or PEX (for example, see Specification F 1986 or F 1281). This test method outlines the requirements of a pressurized flow-through test system, test pressure, 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 the most common system in use today. Disinfecting systems other than chlorine have not been evaluated by this method.

1.2 This test method is valid for polyolefin materials that are stabilized with hindered phenolic type anti-oxidants that shall be analyzed using the Oxidative Induction Time (OIT) method (for example, D 3895).

1.3 Guidelines and requirements for test temperatures, test hoop stresses, and other test criteria shall be established by prior testing of multilayer tubing and solid-wall PEX and PE tubing. Other related system components that typically appear in a multilayer tubing hot-and-cold water distribution system shall be evaluated with the multilayer tubing. When testing multilayer tubing 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 affect 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), polyethylene (PE), polypropylene (PP), cross-linked polyethylene (PEX), and multilayer (polymer-metal composite) pipes.

1.4 This test method is applicable to multilayer tubing 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.

1.5 The values stated in cm-gram units are to be regarded as the standard. The values given in parentheses are for information purposes.

1.6 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

D 1600 Terminology for Abbreviated Terms Relating to Plastics

D 2122 Test Method for Determining Dimensions of Thermoplastic Pipe and Fittings

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

F 412 Terminology Relating to Plastic Piping Systems

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminology F 412 and abbreviations shall be in accordance with Terminology D 1600, unless otherwise indicated.

3.1.1 *ductile failure (Stage I), n*—failure in the tubing wall that is characterized by obvious localized deformation of the material visible with the unaided eye. Ductile failures produced with this test method shall not be used for data analysis.

3.1.2 *brittle failure (Stage II), n*—failure in the tubing 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 tubing to the outside surface typically resulting in a pinhole leak. Brittle failures produced with this test method shall not be used for data analysis.

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.

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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.

3.1.3 *environmental or oxidative failure (Stage III), n*—failure in the tubing wall characterized by a large number of cracks emanating from the interior surface of the tubing wall.

3.1.3.1 *Discussion*—Stage III failures shall be permitted to be identified by a color shift in the failure area (typically brown or reddish-brown). Identification of oxidative failure, when not obvious by inspection with the unaided eye, can be performed with a 25× microscope or other similar device yielding the same level of magnification. Only Stage III environmental failures shall be used for data analysis.

3.1.4 *hot-and-cold water distribution system, n*—a combination of components such as tubing, fittings, valves, and so forth, that when installed as a complete system, make up the interior water supply system of a commercial or residential structure.

3.1.5 *multiple linear regression, n*—a three or four coefficient mathematical model used to analyze time-to-zero OIT data from different temperatures to extrapolate projected time-to-zero OIT at selected temperatures.

3.1.6 *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 F 2023.

4. Summary of Test Method

4.1 The multilayer tubing or tubing/fitting assemblies are exposed to pressurized test-fluid for sufficient times to generate multiple OIT values in Regime A and B.

5. Significance and Use

5.1 Environment or oxidative time-to-zero OIT data derived from this test method, analyzed in accordance with Section 13, shall be suitable for extrapolation to typical end-use temperatures and time. The extrapolated value(s) provides a relative indication of the resistance of the tested multilayer tubing or system to the oxidative effects of hot, 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 hot-and-cold water distribution system that are not addressed in this test method. As such, the extrapolated values do not constitute a representation that a multilayer tube or system with a given extrapolated time-to-zero OIT value shall perform for that period of time under actual use conditions.

NOTE 3—the extrapolated value of the oxidative induction time-to-zero OIT data provides a conservative estimate of the life of the multi-layer tube.

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.

6.1.1 *Recirculating Test System*—A flow-through test system that repeatedly reconditions the test-fluid and passes it through the specimens. For purposes of this test method, the test-fluid shall be monitored at a sufficient frequency to ensure that it continuously meets the test-fluid parameters and water quality criteria.

6.1.2 *Single-Pass Test System*—A flow-through test system that passes the test-fluid through the specimens only once and is discarded.

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 Conditions

7.1 *Sampling*—Select at random, a sufficient amount of tubing 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 multilayer tubing specimens shall be 200 to 300 mm (8 to 12 in.) in length between fitting closures or between fitting joints. The nominal tubing size (diameter) shall be the size within the product line which has the thinnest inner wall layer thickness.

7.3 *Testing as a System*—When testing multilayer tubing and related system components (such as fittings) as a system, the other components shall be attached to the multilayer tubing 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 Data*—A minimum of forty-two (42) data points is required. A data point is the OIT measurement taken after an exposure time, t . Statistical reliability of the analysis of the resultant data shall be improved by obtaining additional data points at each temperature/regime condition.

7.4.1 *Test Data Distribution*—OIT data points shall be obtained for at least seven exposure times at each of a minimum of 3 test temperatures. No more than three OIT measurements shall be used from samples at each exposure time in generating the minimum of 42 data points. At each temperature there shall be a minimum of 6 OIT data points in Regime A and 8 OIT data points in Regime B.

NOTE 4—For initial testing of a new material it is recommended that sufficient samples be started testing to accommodate at least 12 exposure times at each temperature. This will help to improve the accuracy of the transition point from Regime A to Regime B as well as the extrapolation of Regime B data.

7.4.2 *Test Temperature Selection*—Temperatures of 115°C (239°F), 105°C (221°F), and 95°C (203°F) have been utilized in prior testing of PEX. Temperatures of 105°C (221°F), 95°C (203°F), and 80°C (176°F) have been utilized in prior testing of PE. Adjacent test temperatures shall be separated by at least 10°C (18°F). Other test temperatures shall be permitted to be

used, but the maximum test temperature shall not exceed 120°C (248°F) for PEX or PP and 115°C (221°F) for PE.

7.4.3 *Test Pressure*—The specimens shall be subjected to an internal hydrostatic pressure of 0.55 MPa (80 psig) for all temperatures.

7.5 *Oxidative Induction Time (OIT) Specimens*—The specimens for OIT measurement shall be approximately 20 mm (0.8 in.) in length, taken from the inner layer at the approximate midpoint of the pipe length. When conducting multiple OIT measurements from one pipe sample, the specimens shall be spaced evenly along the pipe length, and shall be taken a minimum of 2 pipe diameters from the end of the pipe sample. The inner layer specimen shall be obtained by removal of the outer layers with the use of a lathe or similar tooling. Individual OIT specimens shall be approximately 6.0 mm (0.24 in.) in diameter, so as to fit into the DSC sample pan. These discs are punched out of the specimens prepared above. Care shall be taken to insure that no adhesive layer, if present, is included in the OIT specimen.

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 7.0 and contain 3.0 ppm to 5 ppm (milligrams per liter) of free-chlorine. The chosen pH shall be maintained to ± 0.2 and the chosen free-chlorine concentration shall be maintained to ± 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 $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) of the test temperature.

9.1.3 *Pressure Control*—The pressure of the test fluid shall be maintained to ± 20.69 kPa (± 3 psig).

9.1.4 *Test Fluid Flow Rate*—The flow rate of the test fluid shall yield a minimum velocity of 0.04 mps (0.12 fps). For the nominal size 1/2-in., SDR9 tubing, this corresponds to a flow rate of 0.23 lpm (0.06 gpm). The formula used to calculate the flow rates for other sizes and DR's is as follows:

$$lpm = (MPS) \pi (id/2)^2 (60/1000) \quad (1)$$

where:

id = measured inside diameter of the tubing, mm,

MPS = average flow velocity, m/s, and

lpm = volumetric flow rate, lpm.

9.2 Test Fluid Instrument Accuracy:

9.2.1 *pH*—The pH measurement and control instruments shall have an accuracy of 0.1 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 $\pm 2.5^\circ\text{C}$ ($\pm 4.5^\circ\text{F}$). Direct, forced-air heating of the specimens shall not be prohibited.

11. Specimen Positioning

11.1 The specimens shall be positioned vertically or horizontally. Horizontal positioning requires special attention to ensure 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 *Dimensions Measurement*—Measure and record the critical dimensions for tubing and fittings. For tubing, measure the average outside diameter and wall-thickness (inner and outer layers, metal layers, adhesive layers). For fittings, measure those dimensions critical to the function of the joint, as well as minimum body wall thickness.

12.2 Conduct OIT measurements in triplicate on the inner layer from one specimen per D 3895. The PEX specimen shall be placed in the crucible with the innermost layer (water contact side) face up. The isothermal temperature for the OIT test shall be 190°C, or the results shall be shifted to 190°C using the Arrhenius equation with parameters (for example, activation energy) known for the material being tested. Record these values as the OIT at time 0 h.

12.3 Determine basic properties of the inner layer material.

12.3.1 Where the inner layer material is PEX, conduct gel content analysis of the inner layer of PEX from the specimen used in 12.2 per D 2765, Method B, with the only deviation being that the material shall be a strip machined from the inner layer with a strip thickness of 0.1 mm (0.004 in).

12.3.2 Where the inner layer material is PE or PP, determine the density and melt index.

12.4 Connect 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.

12.5 Maintain the test condition for the number of hours required for the next OIT data points.

12.6 Remove one specimen when the exposure time is reached. Dry and store the specimen at $23 \pm 2^\circ\text{C}$ ($73 \pm 3^\circ\text{F}$) for at least 16 h prior to conducting the OIT measurement(s).

12.7 Conduct the OIT measurement(s) per D 3895 on the inner layer only. The specimen shall be placed in the crucible

with the innermost layer (water contact side) face up. The isothermal temperature for the OIT test shall be 190°C, or the results shall be shifted to 190°C using the Arrhenius equation with parameters (for example, activation energy) known for the specific material being tested. Record the OIT value(s) and exposure time and temperature for this specimen. See Appendix X1 for an example of shifting OIT data to 190°C.

12.8 Repeat steps 12.4-12.7 until the data requirements of 7.4—Minimum Required Data have been met.

13. Calculation

13.1 *OIT Data Analysis*—Determine the Regime A and B regression equations in accordance with 13.1.1-13.1.3.

13.1.1 *Shift OIT Data to 190°C*—For OIT data generated at an isothermal temperature other than 190°C, shift the times to 190°C using the Arrhenius relationship specific to the material being test.

13.1.2 *Convert Exposure Times*—The OIT data is evaluated as a function of root exposure time ($h^{1/2}$) using linear least squares regression. Where the computer software being used to calculate the regression coefficients will not convert this directly, the exposure time must be converted to root-hours before using in the program.

13.1.3 *Determine the Regression Equations*—Determine the linear least-squares regression equation at each temperature for Regime A and Regime B. See Appendix X2 for an example calculation. Where some of the data points occur near the transition between Regimes, statistical methods such as minimum residual variance shall be permitted to be used to determine which data points shall be attributed to each regime.

13.2 *Extrapolations*—Estimate the time to zero OIT at 60°C and 82°C, and the estimated temperature to zero OIT at 50 years in accordance with 13.2.1-13.2.3. See Appendix X3 for an example of these calculations.

13.2.1 *Extrapolated Time-to-Zero OIT at Test Temperatures*—Calculate the extrapolated time-to-zero OIT at each test temperature by setting OIT = 0 h in the regression equation for Regime B at each temperature:

$$t_{T1} = (-b_{T1} / m_{T1})^2 \quad (b, m \text{ are intercept and slope of Regime B equation for temperature } T1)$$

$$t_{T2} = (-b_{T2} / m_{T2})^2 \quad (b, m \text{ are intercept and slope of Regime B equation for temperature } T2)$$

$$t_{T3} = (-b_{T3} / m_{T3})^2 \quad (b, m \text{ are intercept and slope of Regime B equation for temperature } T3)$$

13.2.2 *Linear Regression of Extrapolated Zero OIT Times*—Determine the regression equation of the logarithm of the extrapolated times-to-zero OIT (t_{T1} , t_{T2} , t_{T3}) versus inverse temperature ($1/T$), based on an Arrhenius relationship:

$$\text{Log}(t) = \text{Log}(A) + \Delta E / (2.303R)(1/T) = b_0 + b_1/T \quad (2)$$

where:

- t = time, h,
- T = temperature, K,
- A = constant,
- ΔE = activation energy, J/mol,
- R = universal gas constant, 8.314 J/mol·K,
- 2.303 = conversion factor for using base10 logarithm rather than natural logarithm, and
- b_0, b_1 = regression coefficients.

13.2.3 *Extrapolated Time-to-Zero OIT*—Using the coefficients b_0, b_1 found in 13.2.2, calculate the extrapolated time to zero OIT at 60°C (333K) and 82°C (355K).

13.2.4 *Estimated Temperature at 50 Years*—Using the coefficients b_0, b_1 found in 13.2.2, invert the equation and calculate the extrapolated temperature resulting in a zero OIT time of 438 000 h:

$$T, ^\circ\text{C} = [b_1 / (\text{Log}(438\,000) - b_0)] - 273 \quad (3)$$

This estimated temperature shall be sufficiently lower than the maximum test temperature to allow for a 50-year extrapolation per ISO 9080. Where not, then either the 50-year temperature for the product shall be lowered, or additional testing at a higher temperature shall be conducted.

14. Report

14.1 *Report Content*—Report the minimum information as required in 14.2-14.8.

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 multilayer tubing in the report shall include: tubing nominal size and DR or wall thickness specification; average outside diameter and minimum inner layer wall thickness of each specimen; tubing manufacturer's name, trade designation and tubing lot number (if applicable); resin manufacturer's name and compound designation, and lot designation (for in-plant compounded materials, resin manufacturer's name may be omitted); cross-linking process (for PEX); and gel content (for PEX).

14.5 Identification of fitting(s) tested with the tubing (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 and internal pressure; specimen external air temperature; test fluid free-chlorine concentration and pH; test fluid ORP; water type (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, exposure times, OIT measurements and regime for all specimens tested.

14.8 A summary of the regression analysis including: coefficients; correlation coefficient (R^2 value); estimated time-to-zero OIT at 60°C (140°F); estimated time-to-zero OIT at 82°C (180°F), and temperature at which the estimated time-to-zero OIT is 50 years.

15. Precision and Bias

15.1 *Precision*—The interlaboratory precision of this test method is being determined and will be available on or before January, 2008.

15.2 *Bias*—No information can be presented on the bias of this test method because no material having an accepted reference value is available.

16. Keywords

16.1 antioxidant diffusion; chlorine; composite pipe; multi-layer tubing; OIT; oxidation; oxidative failure; oxidative induction time; PE-AI-PE; PEX-AI-PEX

APPENDIXES
(Nonmandatory Information)
X1. EXAMPLE OF SHIFTING OIT DATA TO 190°C

X1.1 *Rationale*—For test specimens removed at early exposure times and/or being run at the lower temperatures, the OIT test can take significantly longer to run than later specimens or higher temperatures. In those cases, it may be advantageous to conduct the OIT at a higher isothermal temperature, resulting in shorter test duration. This does, however, involve more data analysis and initial testing, as well

as introducing another variable into the test method. For this reason, it is preferred that all OIT data be run at 190°C unless this is not feasible within the constraints of the testing program.

X1.2 *Example Data*—In the table below, the OIT value is given for 6 different isotherms. This data is then fitted to the Arrhenius relationship utilizing the plot shown in Fig. X1.1.

TABLE X1.1 Example Data Set for OIT Shift

Determination of activation energy of unexposed sample						
$\text{Ln(OIT)} = K + \Delta E/R (1/T)$						
R = 8.314 J/mol-K						
Temp, C	OIT, min	1/T	ln(OIT)	K	ΔE , kJ/mol	R ²
230	25	0.001987	3.219			
220	36	0.002028	3.584	-25.19	118.0	0.975
210	52	0.002070	3.951			
200	105	0.002113	4.654			
190	255	0.002159	5.541			
185	376	0.002183	5.930			

Determination of activation energy
slope = $\Delta E/R$

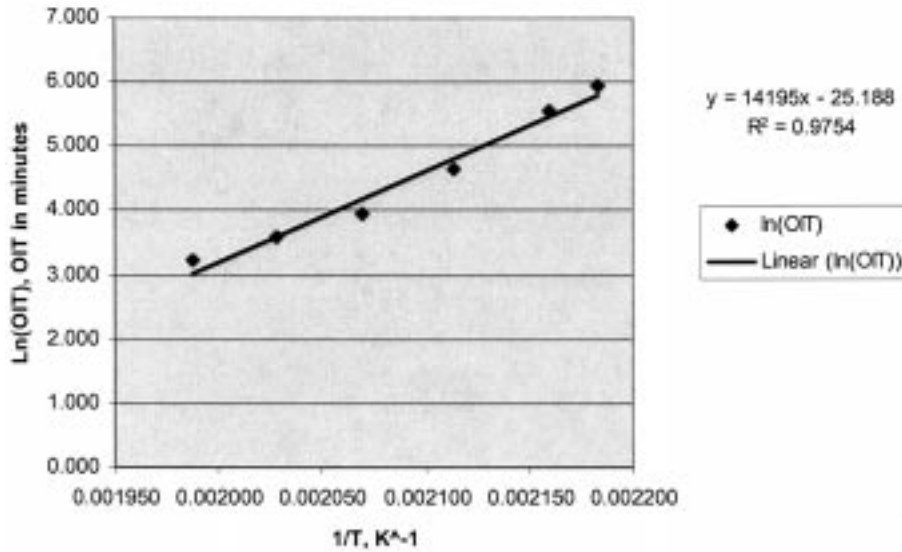


FIG. X1.1 Plot of Ln(OIT) versus 1/T

X2. EXAMPLE REGRESSION FOR REGIMES A AND B

TABLE X2.1 Linear Regression for Regimes A and B at Each Temperature

Calculate transition point and OIT = 0 intercept for Regime B data at each temperature
OIT = $m\sqrt{t} + b$

Temp, C	Regime	Slope, m	Intercept, b	Correlation, R ²	Transition, (h) ^{1/2}	A-B Transition Time, h	OIT = 0, (h) ^{1/2}	Exposure Time at OIT = 0, h
115	A	-1.710	49.22	0.968				
	B	-0.185	10.33	0.698	25.5	651	55.7	3103
105	A	-0.808	45.13	0.943				
	B	-0.230	19.70	0.645	44.0	1940	85.5	7312
95	A	-0.569	50.16	0.989				
	B	-0.104	16.61	0.714	72.2	5220	159.3	25391

X3. EXAMPLE EXTRAPOLATION CALCULATIONS

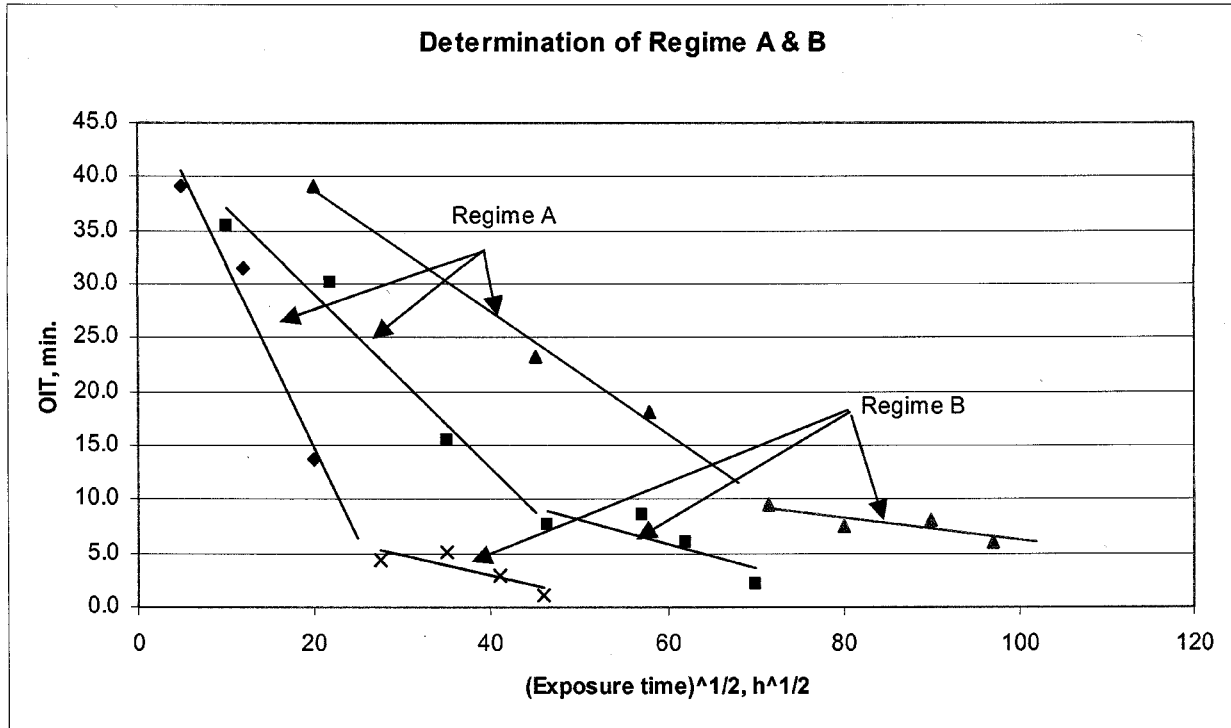


FIG. X2.1 Example of Data at 3 Temperatures for Regime A and B

TABLE X3.1 Example of Extrapolation

Linear regression of OIT = 0 hydrostatic times versus 1/T

$$\ln(t) = \ln(a) + b(1/T)$$

$$\text{Log}(t) = \text{Log}(a) + b * \text{Log}(e) * (1/T) = A + B/T$$

T, °C	1/T, K ⁻¹	t at OIT = 0	ln(t)	ln(a)	b	a	A = Log(a)	B = b*Log(e)
115	0.002576	3103	8.040	-30.77	15040.8	4.3468E-14	-13.362	6532.13
105	0.002644	7312	8.897					
95	0.002716	25391	10.142					
Extrapolations:			Temp	Time, h	Time, yr			
			60	1759291	201			
			82	107339	12			
			70.7	438000	50			

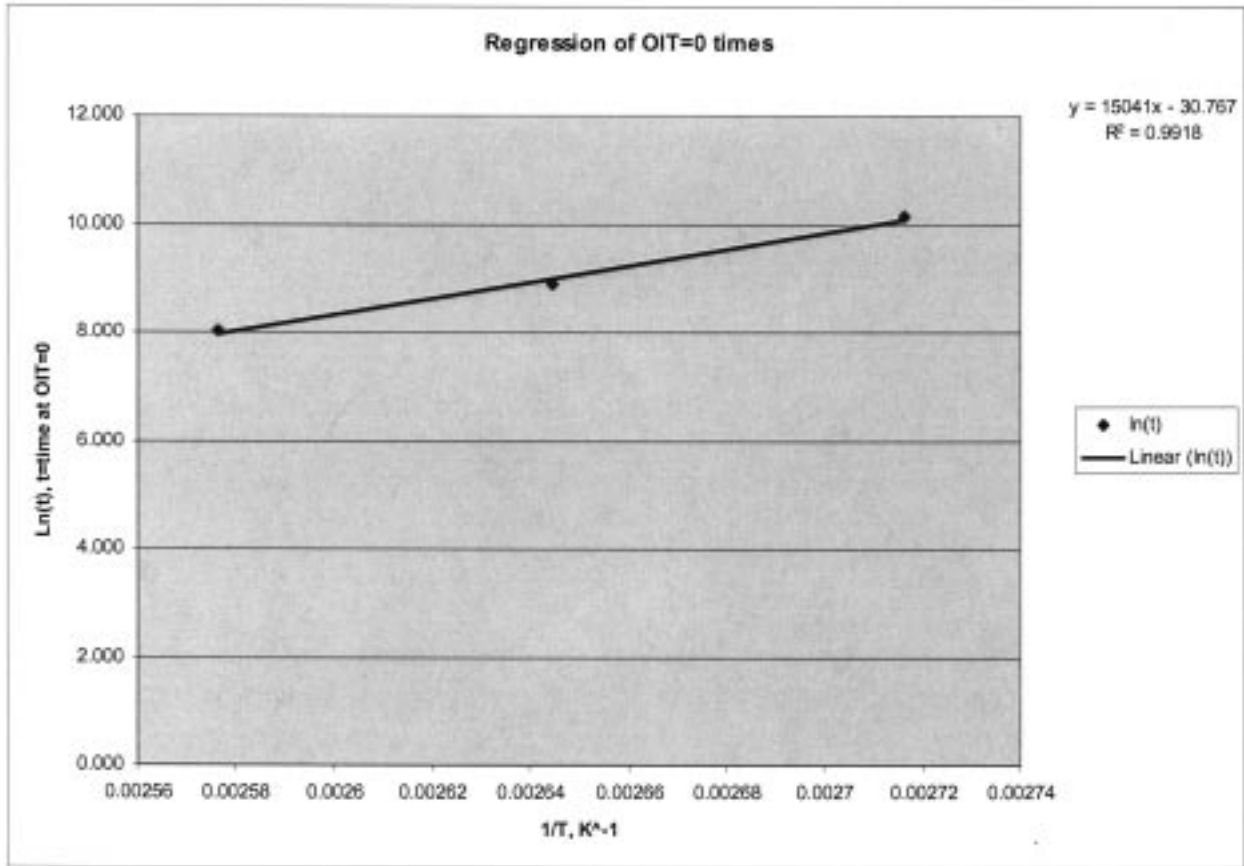


FIG. X3.1 Linear Regression of Extrapolated Fail Times

X4. REFERENCES FOR THE ANTIOXIDANT DIFFUSION MODEL

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(2) Karlsson, K., Smith, G., and Gedde, U., “Molecular Structure, Morphology and Antioxidant Consumption in Medium Density Polyethylene Pipes in Hot-Water Applications,” Studsvik AB, *Polym. Engr. Sci.*, 1991.

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(4) Viebke, J., Hedenqvist, M., and Gedde, U., “Antioxidant Efficiency Loss by Precipitation and Diffusion to Surrounding Media in Polyethylene Hot-Water Pipes,” *Polym. Engr. Sci.*, 1996.

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