FLORIDA DEPARTMENT OF TRANSPORTATION
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FLORIDA METHOD OF TEST FOR PREDICTING THE OXIDATION RESISTANCE OF HDPE CORRUGATED PIPES

Designation, FM 5-574

1. SCOPE

1.1 This test method is used to predict the oxidation resistance of corrugated high density polyethylene (HDPE) pipes in view of Florida DOT 100-year design service life requirement. This protocol utilizes the oxidative induction time (OIT), tensile and melt index tests to evaluate accelerated aging pipe samples.

1.2 The age acceleration is achieved by incubating at a minimum of three different elevated temperatures in a water environment.

1.3 The OIT is used to assess the remaining functional antioxidants in the aging samples.

1.4 The melt index test is used to assess changes in the molecular weight of the aging samples.

1.5 The tensile test is used to assess changes in the engineering properties of the aging samples.

1.6 The tensile and melt index tests are performed along with OIT test to monitor changes in the antioxidant and polymer structure during the course of incubation

1.7 The three sets of data obtained from the elevated temperatures are extrapolated to a lower site specific temperature using the Arrhenius equations to determine the depletion rate of antioxidants, to predict the lifetime of the antioxidant package and to predict the lifetime of the pipe sample.

2. REFERENCED DOCUMENTS

2.1 ASTM Standards:

D1600 Terminology for Abbreviated Terms Relating to Plastics

D3895 Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry

D1238 Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer

D638 Test Method for Tensile Properties of Plastics

2.2 FDOT Documents:

FM 5-572 Test Method for Determining Slow Crack Growth Resistance of HDPE Corrugated Pipes.

3. INCUBATION PROCEDURE

3.1 Procedure A – Single Point Test

3.1.1 Incubate three junction specimens according to ***FM 5-572, Procedure B***.

3.1.2 The temperature of the water bath shall be maintained at 80 ± 1ºC.

Note 1: Other temperatures can be used with the agreement of parties involved.

3.1.3 A tensile stress of 250 psi shall be applied to each specimen.

3.1.4 The duration of the incubation shall be 195 days.

3.1.5 The OIT value of each of the three incubated specimens shall be evaluated using test procedure according to ***Section 4.1.1***.

3.2 Procedure B – Full Oxidation Resistance Test

3.2.1 Prepare a minimum of 45 pipe samples (liner and crown) with dimensions of 5 inches in the circumferential direction and 4 inches in the longitudinal direction.

3.2.2 Place 15 samples in each hot water bath.

Note 2: Since polyethylene is lighter than water, stainless steel metal clips shall be attached to the samples to hold them down. The samples must be separated from each other during the incubation.

3.2.3 The incubation shall be at three different temperatures with a 10ºC interval between them. The temperatures of the three baths shall range between 65 and 85ºC.

Note 3: Four different temperatures will generate greater accuracy in the extrapolation. In this case the lowest temperature shall be 55ºC.

3.2.4 In the first 3 months, incubated samples shall be removed every month for evaluation; thereafter remove an incubated sample from each of the baths every 3-months.

3.2.5 The duration of the incubation shall be different depending on the intended scope of the test.

3.2.6 For predicting the lifetime of the antioxidant package, the incubation shall be carried out until an 80% decrease in the OIT value at the two higher temperatures and a 60% decrease at the lowest temperature are seen.

3.2.7 For predicting the lifetime of the pipe, incubation shall be carried out until there is an 80% decrease in the tensile breaking strain for all incubation temperatures.

4. EVALUATION OF ORIGINAL AND INCUBATED SAMPLES

4.1 OIT Test

4.1.1 Perform OIT tests according to ***ASTM D3895***, employing the following procedures:

4.1.1.1 Use an open aluminum pan.

4.1.1.2 A two-point temperature calibration must be performed once a week.

4.1.1.3 A single test shall be performed on each of the three samples incubated in Procedure A. Two replicates shall be tested for incubated samples in Procedure B.

4.2 Melt Index (MI) Test

4.2.1 Perform MI tests according to the ***ASTM D 1238*** using the test condition 190ºC/2.16 kg.

4.2.2 Two replicates shall be tested for the three incubated samples in Procedure A.

4.3 Tensile Test

4.3.1 Five ***ASTM D 638-TypeV*** test specimens shall be die cut from the original non-incubated sample and incubated samples. The length of the specimens shall be parallel to the longitudinal direction of the pipe.

4.3.2 Perform tensile tests according to ***ASTM D638-TypeV***, using a strain rate of 2 in/min. Record yield stress, yield strain, break stress and break strain.

Note 4: The yield strain and break strain can be obtained using cross-head movement instead of an extensometer.

5. DETERMINE ANTIOXIDANT DEPLETION RATE

5.1 To perform the analysis, the OIT value at two of the three incubation temperatures much reach greater than 80% reduction (20% retained) and 60% for the lowest temperature.

5.2 Plot the average OIT retained value in ln(OIT) versus incubation time, as shown in Figure 1.

5.3 Fit the data with the exponential equation starting from 100% OIT retained.

5.4 The slope of the line is the depletion rate of the antioxidant at that test temperature.

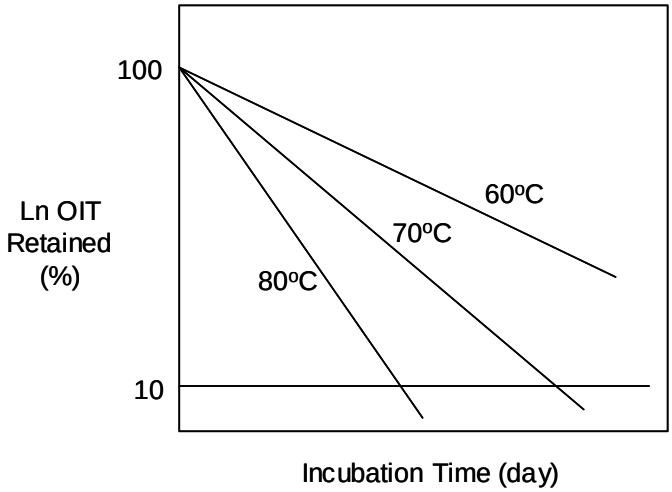

Figure 1 – Ln(OIT) versus time plot

Note 5: Depending on the antioxidant formulation of the evaluated pipe, the OIT data may not be fitted with an exponential equation, as shown in Figure 1. A bi-linear model may be used as illustrated by Viebke and Gedde (1997)[1].

6. ANTIOXIDANT LIFETIME PREDICTION

[1] Viebke, J. and Gedde, U.W. (1997), "Antioxidant Diffusion in Polyethylene Hot-Water Pipes", Polymer Engineering and Science, Vol. 37, No. 5, pp. 896-911.

6.4 To perform the prediction analysis, the OIT value at two of the incubation temperatures much reach greater than 90% reduction (10% retained) and 80% for the lowest temperature

6.5 Plot the average OIT retained value in (OIT) versus incubation time, as shown in Figure 1.

6.6 Fit the data with exponential equation starting from 100% OIT retained.

6.4 Present the slope (S) values in a table together with the incubation temperature as shown in Table 1.

Table 1 – Antioxidant depletion rate at each incubation temperature

Slope (S)	Incubation Temperature (T) (°C)	Incubation Temperature (T) (K)	Inverts Temperature (1/T) (1/K)

6.5 Perform Arrhenius plot by plotting ln(S) versus (1/T). The three data points shall be fitted with a straight line, as shown in Figure 2.

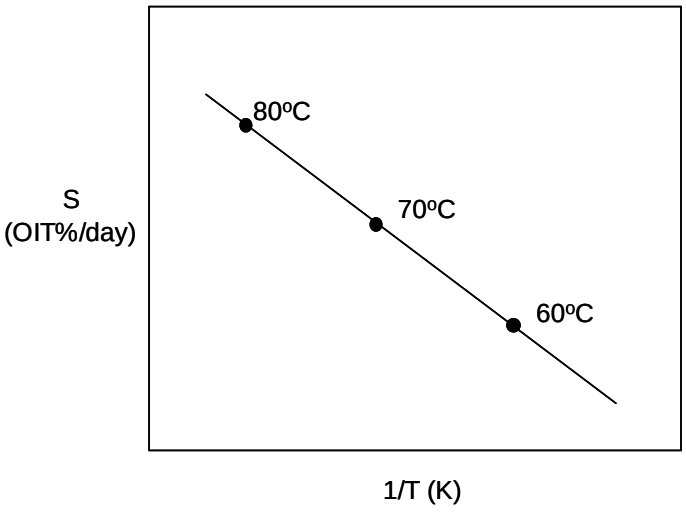

Figure 2: Arrhenius plot of the antioxidant depletion rate versus temperature

6.6 The resulting Arrhenius equation, as shown in Eq. (1), from Figure 2 shall be used to extrapolate the antioxidant depletion rate (S) at specific site temperature.

$$S = A*\exp(-E/RT) \quad (1)$$

where:

S = OIT depletion rate
E = Activation energy of the antioxidant depletion reaction under this test condition (kJ/mol)
R = gas constant (8.314 J/mol.K)
T = test temperature in absolute temperature (K)
A = constant

6.7 The average temperature of 23°C shall be used as the general site temperature in the lifetime extrapolation analysis.

6.8 The lifetime (t) of the antioxidants at site specific temperature shall be calculated using Eq. (2).

$$OIT = P* \exp(- S*t)$$

where:

OIT = critical OIT time (min.)
P = original OIT of the material (min.)
S = OIT depletion rate (min/day)
t = lifetime (days)

Note 6: The critical OIT value can be corresponded to the onset of a decrease in tensile elongation, the onset of an increase in MI value, or the OIT value of unstabilized polyethylene pipe resin.

7. PREDICTION METHOD TO DETERMINE LIFETIME OF PIPE BASED ON OXIDATION DEGRATION

7.1 To perform this analysis, the break strain shall decrease more than 80% for all three incubation temperatures.

7.2 Calculate the average break strain at each incubation interval. Determine the percent break strain retained value using Eq. (3).

$$\%\ break\ strain\ retained = \frac{average\ strain\ value\ at\ each\ incubation\ interval}{average\ strain\ value\ of\ original\ sample} \quad (3)$$

7.3 Plot the percent break strain retained value versus incubation time for three incubation temperatures, as shown in Figure 3. From the plot, determine the time to reach 20% break strain retained (i.e., 80% drop in break strain).

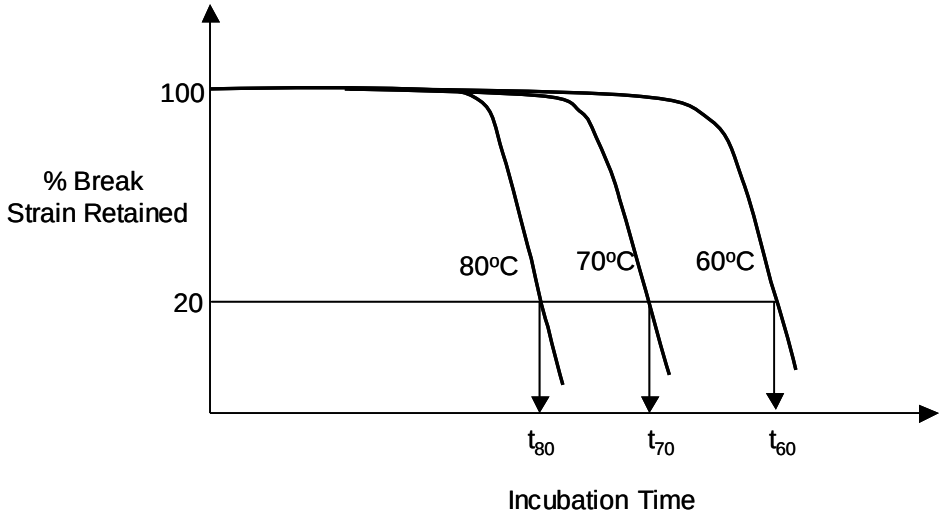

Figure 3 – Determine the time to reach 20% break strain retained

7.4 Determine the reaction rate at each incubation temperature using Eq. (4).

$$\text{Reaction Rate}\,(R_{\text{temperature}}) = \frac{1}{t_{\text{temperature}}} \qquad (4)$$

7.5 Perform Arrhenius plot by plotting ln($R_{temperature}$) versus ($1/T$). The data shall be fitted with a straight line.

7.6 The Arrhenius equation obtained from 6.5 shall be used to extrapolate the 80% drop in break strain at specific site temperature (t_{site}), which is the lifetime of the pipe based on oxidation degradation.

8. REPORTING RESULTS

8.1 Procedure A

8.1.1 Report the three individual OIT values.

8.1.2 Report the average and the minimum OIT of the three values.

8.2 Procedure B

8.2.1 The material properties versus incubation time plot for all three incubation temperatures.

8.2.2 Report antioxidant depletion rate at each incubation temperature.

8.2.3 Report lifetime of antioxidant in years.

8.2.4 Report lifetime of the pipe based on oxidation degradation in years.