Standardization of Oxidation Induction Time Testing Used in Life Assessment of Polymeric Electric Cables

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ABSTRACT: Oxidation induction time (OIT) has been proven to be a useful diagnostic tool in assessing the extent of degradation in the polymer insulation of electric cable in nuclear power plants. Factors that influence OIT include test temperature, sample preparation, sample geometry, sample mass, particle size, thermogram interpretation, and shelf life. The effects of these parameters were investigated for ethylene propylene diene monomer (EPDM) and crosslinked polyethylene (XLPE) polymer cable insulations, which were aged in radiation and thermal environments. The results were then used to recommend standards for an OIT methodology suited for practical use in the nuclear power industry. Techniques to estimate error in thermogram interpretation and reproducibility were also developed. © 1997 John Wiley & Sons, Inc. J Appl Polym Sci **66:** 1691–1702, 1997

Key words: oxidation induction time; OIT; OIT standardization; cable monitoring; cable aging; cable life assessment

INTRODUCTION

Oxidation induction time (OIT) testing has been proposed as a useful method to measure aging of electric cable in nuclear plower plants.[1] The OIT of a polymer sample is measured using a differential scanning calorimeter (DSC) in the isothermal mode as the time from oxygen introduction until the onset of rapid oxidation of the sample. Rapid oxidation occurs after the antioxidant of the sample has been exhausted. Thus, the OIT is related to the amount of antioxidant in the sample at the beginning of the test. As a polymer ages during its normal use, the antioxidant in the polymer decreases so that the OIT of the polymer also decreases, making OIT a measure of aging.

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General ASTM standards exist pertaining to OIT testing.[2,3] However, these standards are somewhat general. More specific standardized methodology is needed for accurate aging assessment of low-voltage cable insulation used in nuclear power plants. Experimental results leading to specific recommendations for standardized OIT testing of cable are presented here. A comparison of the methods recommended here to applicable ASTM standards is shown in the Appendix.

Standardization requires detailed investigation into a variety of factors that influence OIT. The primary factors, or parameters, that affect OIT can be categorized as either instrumentation or sample effects. Instrumentation effects include DSC test temperature and thermogram interpretation. Polymer sample effects include preparation type (e.g., grinding versus slicing), geometry, particle size, mass, and shelf life. The research reported here is an experimental investigation of these factors. Further details appear in Mason and Reynolds,[4] and Mason.[5]

Stresses inside a nuclear reactor containment to which cables are exposed during normal operation include radiation, thermal, mechanical, and electrical stresses. These cables are qualified to withstand radiation and thermal exposures that emulate normal operation and postulated accidents in accordance with industry guidelines.[6,7] The stresses studied in this research are limited to radiation and thermal, which are the stresses which dominate the aging of lower voltage cable polymers. Earlier measurements of OIT as a function of radiation aging are reported in Bell et al.,[8] Mason et al.,[9] and Anandakumaran et al.[10]

The polymers used in this study were low-voltage electric cable insulation used for control and instrumentation functions in nuclear power plants. The two insulations studied were the two most widely used as Class 1E cable for nuclear use. The classifications are ethylene propylene diene monomer (EPDM) and crosslinked polyethylene (XLPE).

The EPDM6G and EPDM7J were two different cable compositions made by the same manufacturer. The numbers 6 and 7 refer to the EPDM batch, G refers to the green cable color, and J refers to a cable that has a thin jacket of Hypalon™. EPDM7J has a Hypalon jacket bonded to it, which was not removed during the aging and OIT measurements; EPDM6G has only EPDM insulation with no bonded jacket. The XLPE polymers were labeled XLPE8B and XLPE10, which were made by different manufacturers. The B refers to the blue cable color, and all of XLPE10 was gray. All cables were qualified for use in nuclear power plants. The cable sizes were AWG 14.

OIT MEASUREMENT

OIT measurements were performed with a Perkin-Elmer differential scanning calorimeter DSC-7. Samples were encapsulated in an aluminum pan with a stainless steel screen lid mechanically crimped in place. Samples were immersed in a steady flow of nitrogen gas while being heated to the isothermal test temperature. When the test temperature was reached, oxygen replaced the nitrogen purge gas.

The OIT is determined graphically from a DSC thermogram. The instrumentation used in this research was a Perkin-Elmer thermal analyzing system TAS 7. Figure 1 illustrates a typical thermogram for use as reference. The y axis is the differential heat flow needed to maintain the reference and sample at the isothermal temperature. The exotherm diverges from the horizontal baseline when the antioxidant is consumed, after which time the actual polymer burns in an exothermic reaction. The OIT is then defined as the time between the introduction of oxygen into the sample housing and the intersection of the baseline extension with the exothermic slope.

STANDARDIZATION FACTORS

Uncertainties in OIT Measurements

Two types of uncertainties are present when measuring the OIT: one is the subjective interpretation of the thermogram, and the other is OIT reproducibility. All OIT errors reported here include both interpretation and reproducibility components.

Although most exothermic slopes give narrow margins for interpretation, some produce larger sources of error. This is caused primarily by the lack of definition in the exothermic slope, which leads to a range of slopes that potentially could be used to intersect the baseline and thus mark the endpoint of the OIT. To standardize this interpretation, the best-fit slope is drawn at both the most rapid and the most gradual rate of change. These two lines are intersected with the baseline, and then the midpoint between the two intersections marks the effective OIT. The interpretation error is reported as the half-width between the effective OIT and the intersection limits. It was observed that aged samples from the same polymer exhibited a strong similarity in curve shapes, which is conducive to consistency in thermogram slope interpretation.

Reproducibility of OIT was measured in the present investigation. The standard deviation for reproducibility, expressed as a fractional error, varies with the value of the OIT. Measurement of reproducibility uncertainty as a function of OIT was accomplished with EPDM6G and XLPE8B at various ages to obtain different OITs, together with two other highly aged cables, EPDM4 and XLPE3. The results are given in Table I, and the fractional errors are plotted on Figure 2 as a function of average OIT. It is noted that there is no appreciable difference in reproducibility between

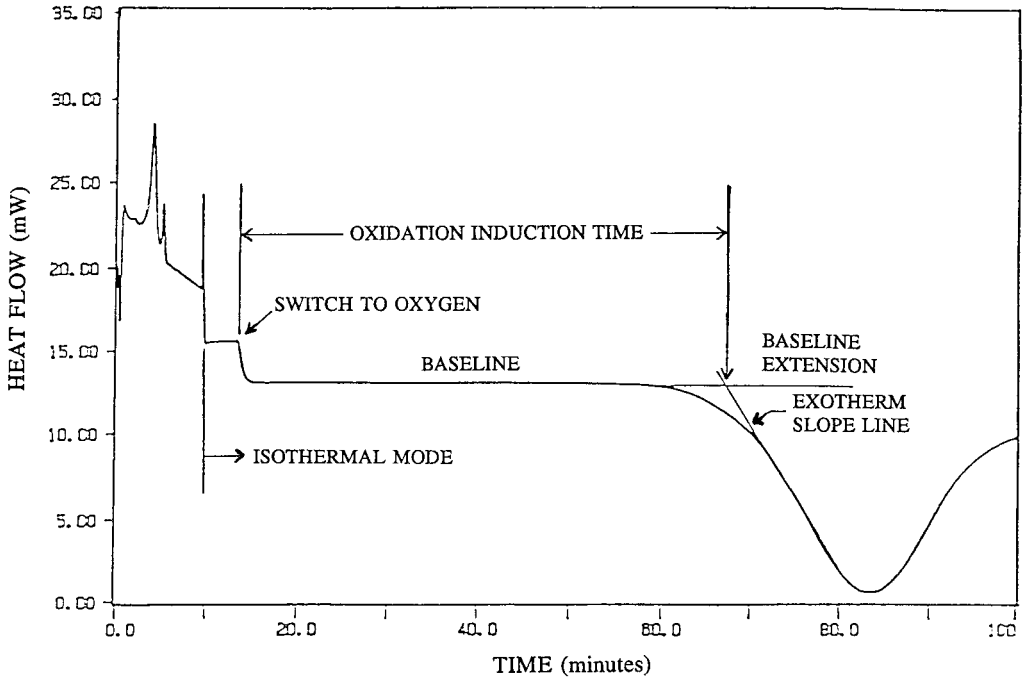

Figure 1 DSC thermogram and illustration of OIT measurement.

EPDM and XLPE. Also the fractional error remains about constant for OITs above 100 min. An error gauge line has been fixed to Figure 2 to determine a conservative reproducibility error for a given OIT.

DSC Test Temperature

OIT has been shown to vary inversely with DSC test temperature. When plotted versus inverse temperature, an Arrhenius correlation is observed. Figure 3 depicts the relationship for several different polymers used in this research. These curves are based on the fundamental equation given by

$$\ln(\mathrm{OIT}) = A + \frac{E_a}{\mathrm{k}T} \qquad (1)$$

where k is Boltzmann's constant, A is the y intercept, E_a is the activation energy, and T is the DSC test temperature. This predictable behavior provides a means by which a suitable DSC temperature can be determined. Two primary factors that govern the DSC test temperature are initial anti-

Table I Reproducibility Errors for EPDM and XLPE

Material	Number of Measurements	Average OIT (min)	Standard Deviation (±min)	Fractional Error[a] (±)
EPDM4	9	11.66	0.77	0.066
EPDM6G	5	60.24	2.28	0.038
EPDM6G	5	97.38	1.96	0.020
EPDM6G	5	175.54	3.05	0.017
XLPE3	9	15.02	1.26	0.084
XLPE8B	5	64.76	1.50	0.023
XLPE8B	5	107.68	1.03	0.010
XLPE8B	5	160.04	2.56	0.016

[a] Ratio of standard deviation to average OIT.

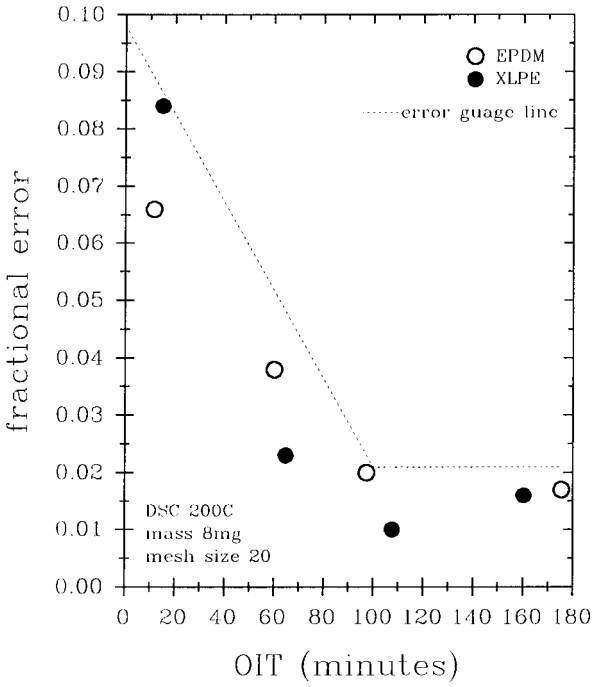

Figure 2 Fractional error (one standard deviation) due to reproducibility of OIT measurements as a function of average OIT. The error gauge line provides a conservative envelope of the data and is the value used for reproducibility errors in individual measurements.

oxidant concentration and range of degradation. A higher initial antioxidant content will produce a longer OIT. This relationship has been shown to increase exponentially in Mason et al.[9] Initial antioxidant concentration (unaged OIT) sets the lower limit on DSC temperature, while the extent of aging (smallest relative OIT) sets the upper limit. The desirable DSC test temperature is therefore specific to the material composition and the aging parameters. For example, an unaged OIT measured at 200°C could be 100 minutes. If an aged polymer has an OIT too short to measure at 200°C, however, this temperature can be lowered to accommodate the evaluation of this heavily degraded sample. The range of DSC test temperatures suitable for nuclear use electric control cables typically ranges from 185 to 225°C.

Sample Preparation

Sample preparation is the means by which the polymeric material is reduced from its whole form to a size compatible with the DSC instrumentation. Two primary methods of preparing are slicing and grinding; the two methods were compared for their effects on the results. Slicing is the method of removing strips from the cable insulation and dicing the strips into small particles (e.g., 20 mesh U.S. Standard Sieve Series). This is achieved using a razor blade and sieve series.[11] Grinding in this research utilized a Wiley Mill to grind the pieces of cable insulation into small particles using the same sieve series for size verification. It has been suggested that the grinding process may cause additional oxidation degradation; hence, this uncertainty was examined.

A particle size of 20 mesh was used to compare the OITs measured with ground and sliced samples for a variety of aging conditions. For 20 mesh, the particle radii lie between 425 and 840 μm. Table II lists the results of these measurements for EPDM and XLPE polymers that were unaged, radiation-aged, or thermally aged. These comparisons were made at the same DSC test temperature, total sample mass, and particle size to isolate the effect of grinding. The observations show that all cables examined have reasonable agreement between sliced and ground particles. The differences are within the uncertainties in Table II. However, a general pattern is observed in that the sliver particle OITs are consistently slightly less than the ground particle OITs. The largest of

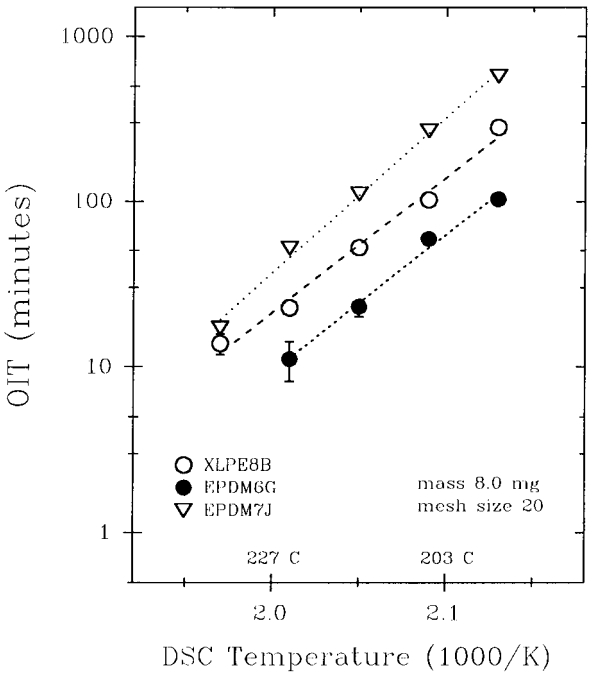

Figure 3 OIT of unaged insulation as a function of DSC test temperature.

Table II Comparison of OIT from Sliver and Ground Samples with the Same Particle Number Density

Material	OIT Ground (min)	Error (±min)	OIT Sliver (min)	Error (±min)
Unaged Sample				
EPDM6G	156	4	151	5
XLPE8B	355	15	344	11
XLPE10	294	6	289	6
Thermally Aged Only				
EPDM6G (5.25 d)	91	4	84	7
XLPE8B (9.75 d)	51	2	47	2
Radiation Aged Only				
EPDM6G (0.3 MGy)	41	3	40	3
XLPE8B (0.3 MGy)	82	3	78	3

these disparities occurs in EPDM6G in which the sliver OIT is 7.7% less than the ground particle OIT. For every other case, the difference is less than 5.0%.

The comparison of ground 20 mesh samples with slivers of comparable size negates speculation that grinding causes significant additional oxidation in the polymer sample, at least for this size of particles. If grinding had caused significant oxidation, the ground particle OIT would have been less than the sliver particle OIT. In addition, the comparison yields information that the difference in OIT of the two methods is virtually negligible, and each means provides an equally accurate OIT measurement. In general, the consistency, reproducibility, and ease of thermogram interpretation prompts the use of ground particles when possible. In the same vein, cable field samples that are scraped from the insulation and sliced into particles provides an equally acceptable means for measuring actual field-aged polymers.

Sample Particle Size

Sample particle size refers to the individual particle size after the reduction method (either grinding or slicing) has been executed. Since particle sizes per unit mass can range from one large particle to many dust-like particles, the effects of this variable should be documented for reliable OIT measurements.

Oxygen must be present in the polymer for oxidation degradation of polymer molecules to occur during a DSC test. The amount of oxygen available within the polymer sample affects the length of the OIT. For oxygen to be present in the sample, it must diffuse into the sample during the DSC test. Particle size affects the extent to which oxygen can diffuse into the sample during a test; for example, the ability for oxygen to diffuse into the sample decreases as the particle size increases. The theory of oxygen diffusion during a DSC test, together with results for 20 to 60 mesh particles, is provided in Mason and Reynolds[4] and Mason.[5]

OIT was measured on samples composed of varying particle sizes. The mass of these samples was held constant at 8.0 mg (a mass recommended by the DSC manufacturer). The particle sizes ranged from one particle of 8.0 mg to 60 mesh size particles that average about 700 particles per 8.0 mg. The range of particle sizes and average particle size for 20, 40, and 60 mesh are given in Table III. 60-mesh particles are about the size of dust, and 20-mesh grains resemble course sand. Particles sizes larger than 20 mesh were cut by hand.

Figure 4 shows OIT as a function of the number of particles for EPDM6G, XLPE8B, and XLPE10 for a constant mass of 8 mg. Two observations can be made from this figure: particle sizes 20 mesh

Table III Mesh Sizes and Corresponding Particle Diameters

Mesh Size	Particle Diameter (mm)
20	0.85–1.680
40	0.50–0.85
60	0–0.50

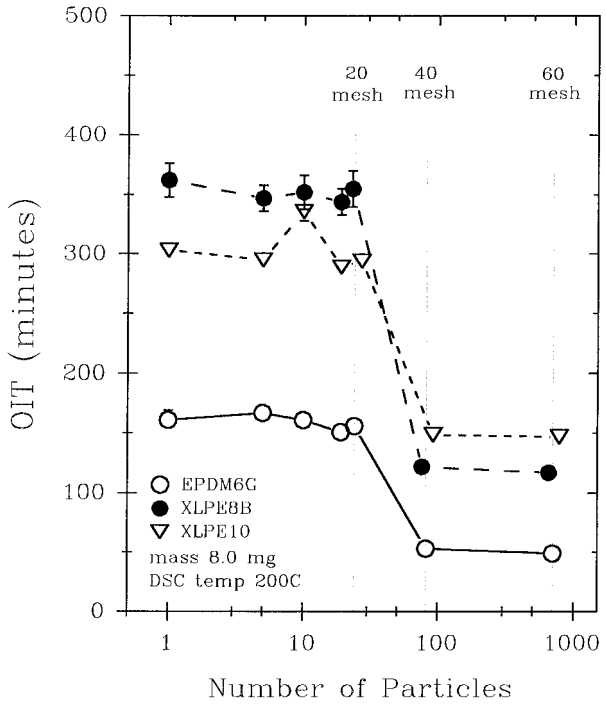

Figure 4 OIT as a function of mesh size and average number of particles for a constant sample mass of 8 mg.

and larger show a relatively constant OIT; and a dramatic drop in OIT is observed at the 40-mesh mark, followed by a leveling off at 60 mesh.

It is recommended that the particle size be standardized at 20 mesh for both ground and sliced particles.

Sample Mass

The effects of sample mass on OIT have been reported for other polymeric materials, including high-voltage cable insulation.[12] The study outlined in this section refers to mass as the total polymer sample mass. The OIT was measured for samples where the mass was varied from 1.0 to 15.0 mg (15.0 mg is the capacity of the sample pan assembly) at a fixed particle size of 20 mesh. Table IV shows the results of these measurements for EPDM6G and XLPE8 polymers.

Observations from these measurements reveal that OIT does not significantly change when the sample mass is varied. The grouping of OIT is within a ±3 minute margin, which is inside the standard deviation generated by thermogram reproducibility and therefore cannot be attributed to the influence of sample mass. The lower mass samples (1.0–3.0 mg) did yield consistently longer OITs than those of higher mass. The primary reason for this subtle trend can be found in the changes that were observed in the exotherm slope.

Figure 5 shows the thermogram curves for several different mass sizes. The curve shape has a strong dependence on sample mass in that as the mass is reduced, the peak of the exotherm is decreased and broadened. This resulted in a less defined peak, which gradually departed from the baseline and forced the OIT intersection to a longer time. However, the pattern was only a subtle phenomenon, and it was further convoluted by the increased difficulty in thermogram interpretation with the broader peaks. This is evidenced by the errors values given in Table IV, which show the highest values for sample masses between 1.0 and 5.0 mg.

The study yielded two primary results: (1) the OIT quantity did not vary significantly with sample mass, but (2) the thermogram curve shape was strongly influenced by sample mass. The shape of the thermogram curve experienced the least change between 7.0 and 10.0 mg. Overall thermogram quality, error margin, and consistency shows the optimum mass range for measuring OIT on nuclear-use polymer cables is between

Table IV OIT as a Function of Sample Mass for XLPE and EPDM[a]

EPDM6G		XLPE8B	
Mass (mg)	OIT (min)	Mass (mg)	OIT (min)
1	39.7 ± 5	1	84.3 ± 5
2	38.4 ± 4	2	83.6 ± 6
3	40.2 ± 5	3	82.9 ± 6
4	37.9 ± 5	4	84.1 ± 6
5	37.9 ± 5	5	82.4 ± 4
6	37.0 ± 4	6	82.0 ± 3
7	36.7 ± 4	7	84.5 ± 3
8	36.8 ± 3	8	81.7 ± 3
9	36.0 ± 3	9	81.3 ± 3
10	36.7 ± 3	10	81.6 ± 2
11	37.9 ± 3	11	85.2 ± 3
12	36.1 ± 4	12	82.8 ± 3
13	36.4 ± 3	13	81.0 ± 3
14	35.3 ± 3	14	84.6 ± 4
15	37.2 ± 3	15	82.6 ± 3

[a] All samples irradiated to 0.3 MGy.

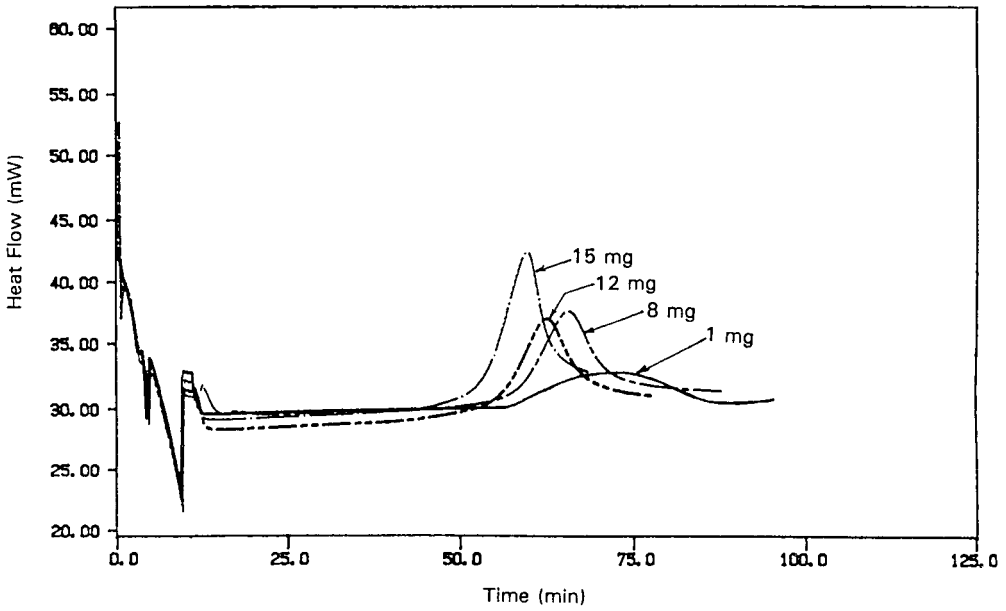

Figure 5 DSC thermograms for different sample masses.

7.0 and 10.0 mg. A standard value of 8.0 mg was employed for this research, which is consistent with the DSC manufacturer's suggested value.

Sample Shelf Life

The potential variation of OIT due to shelf life was examined. OIT measurements within a few days after aging completion were compared with OITs for cable that was stored under different conditions and times.

Storage conditions for this study included temperature, light exposure, sample form (ground or whole), and air exposure. Storage times of two and six months were investigated. Samples were divided into the following storage categories: (1) room temperature, (2) refrigerated, (3) exposed to fluorescent lighting, (4) dark storage, (5) exposed to atmosphere, and (6) sealed air-tight. Samples were stored in both whole form (ground after storage) and granular form (ground prior to storage) for each data point to observe the effects of grinding.

OIT results for EPDM and XLPE are listed in Tables V and VI. The samples were ground to a desired 20 mesh size, and the sample mass was 8.0 mg. Observations from EPDM6G and XLPE8B show a strong retention in OIT after 60 days of storage for all conditions. For EPDM6G, most of the stored sample OITs are within 5% of the unstored value, with one anomaly having an 18% difference (77 compared to 91 min). Likewise, for XLPE8B, the stored sample OITs were mostly within 4% from the unstored value, with one difference noted at 11% (74 min compared to 82 min). Small reductions in OIT occurred during the first two months, but no additional loss was observed between two and six months.

Although nearly all of the differences are within the margin of error, several subtle trends are evident. In general, the highest retained OITs were found in the samples that were sealed and refrigerated in darkness. These OITs were consistently within a few percent of the unstored sample OIT. The lowest OIT retention occurred for samples stored at room temperature in fluorescent light, as might be expected. A comparison between samples ground prior to storage and those ground after storage revealed that the samples ground just before testing exhibited slightly longer OITs.

The investigation showed that the effect of shelf life on the OIT of polymer cables is minimal. Although several subtle trends existed, the margin of difference for nearly every storage condition and time is within the expected error for the OIT. Therefore, the efficacy of OIT measurements after storage is not compromised. Optimum storage

Table V Shelf Life Effects on OIT for EPDM6G as Functions of Storage Time, Aging Stress, and Grinding Sequence

Shelf Storage Mode	Storage Time (months)	OIT (min) Radiation (0.3 MGy; Preground)[a]	Radiation (0.3 MGy; Post-Ground)[b]	Thermal (5 d @ 150°C; Preground)	Thermal (5 d @ 150°C; Post-Ground)
No storage time.	0	36.8 ± 3	36.8 ± 3	91.2 ± 4	91.2 ± 4
Air exposed; room temperature; fluorescent light.	2	31.8 ± 2	33.1 ± 3	77.4 ± 3	84.0 ± 3
	6	29.2 ± 3	32.5 ± 3	77.0 ± 3	84.3 ± 3
Air exposed; room temperature; dark.	2	29.1 ± 3	35.9 ± 3	82.9 ± 4	83.1 ± 2
	6	28.9 ± 3	33.2 ± 4	80.2 ± 4	82.1 ± 5
Air exposed; refrigerated.	2	30.9 ± 3	35.8 ± 4	84.6 ± 4	88.9 ± 3
	6	31.3 ± 2	35.1 ± 4	84.2 ± 3	89.5 ± 3
Sealed; room temperature; fluorescent light.	2	28.5 ± 3	32.4 ± 3	87.3 ± 4	90.8 ± 3
	6	28.0 ± 2	32.6 ± 2	86.9 ± 3	89.9 ± 3
Sealed; room temperature; dark.	2	29.0 ± 2	29.2 ± 3	88.1 ± 3	92.0 ± 3
	6	29.4 ± 2	29.0 ± 3	88.4 ± 5	91.5 ± 4
Sealed; refrigerated.	2	33.4 ± 4	37.3 ± 2	88.9 ± 3	90.3 ± 4
	6	33.0 ± 3	37.3 ± 3	89.0 ± 3	90.7 ± 3

[a] Ground prior to storage.
[b] Ground after storage.

conditions can be loosely postulated as unground, sealed, dark, and refrigerated prior to OIT testing. However, reasonable approximations of the optimal storage conditions will provide satisfactory results. Grinding of stored specimens should occur just prior to OIT testing.

CONCLUSIONS

An experimental investigation of factors affecting the standardization of OIT testing for life assessment of electric cable insulation was completed, leading to specific recommendations for standardization.

Reproducibility in OIT measurements was studied, resulting in a method for assessing the fractional error as a function of OIT. OIT testing was found to be reliable and consistent.

Factors affecting OIT measurement for cable life assessment were identified as DSC test temperature, sample preparation (grinding or slicing), sample particle size, sample mass, and shelf life.

DSC test temperature was found to have an Arrhenius correlation with OIT, which resulted in a practical range of 185 to 225°C, depending on the initial antioxidant concentration and extent of aging.

Preparing samples by slicing or grinding yields virtually no difference in OIT and negated the concern that the grinding process might cause additional oxidation degradation.

OIT was determined to be influenced by the particle size in which sizes of 20 mesh and larger had essentially constant OITs, and the 40 mesh size was marked by a dramatic drop in OIT. Oxygen diffusion was presented as the reason for the reduction in OIT for the smaller particles. A standard particle size of 20 mesh is recommended.

Sample mass affected the OIT in the thermogram peak shape with the smaller masses having a broader and lower peak. However, the actual OIT did not vary significantly between sample

Table VI Shelf Life Effects on OIT for XLPE8B as Functions of Storage Time, Aging Stress, and Time of Grinding

		OIT (min)			
Shelf Storage Mode	Storage Time (months)	Radiation (0.3 MGy; Preground)[a]	Radiation (0.3 MGy; Post-Ground)[b]	Thermal (10 d @ 150°C; Preground)	Thermal (10 d @ 150°C; Post-Ground)
No storage time.	0	81.7 ± 3	81.7 ± 3	50.5 ± 2	50.5 ± 2
Air exposed; room temperature; fluorescent light.	2	74.0 ± 3	77.7 ± 3	47.7 ± 2	48.4 ± 3
	6	73.4 ± 3	75.1 ± 2	46.0 ± 2	47.8 ± 3
Air exposed; room temperature; dark.	2	75.8 ± 3	82.6 ± 4	47.9 ± 2	50.1 ± 3
	6	75.1 ± 3	82.8 ± 3	47.7 ± 2	49.0 ± 3
Air exposed; refrigerated.	2	78.9 ± 3	81.0 ± 3	48.9 ± 3	49.8 ± 2
	6	79.8 ± 3	81.9 ± 3	47.9 ± 3	48.2 ± 2
Sealed; room temperature; fluorescent light.	2	76.8 ± 4	80.3 ± 3	45.3 ± 3	47.9 ± 4
	6	76.2 ± 2	80.0 ± 4	44.1 ± 3	47.3 ± 3
Sealed; room temperature; dark.	2	77.3 ± 3	79.8 ± 3	49.1 ± 2	50.4 ± 3
	6	76.4 ± 3	77.9 ± 3	49.9 ± 4	49.6 ± 3
Sealed; refrigerated.	2	82.1 ± 3	82.3 ± 4	50.1 ± 3	52.0 ± 3
	6	81.3 ± 2	82.0 ± 3	49.4 ± 2	49.8 ± 3

[a] Ground prior to storage.
[b] Ground after storage.

masses. The largest interpretation error was found in the smaller masses, and the optimum mass range was shown to be from 7 to 10 mg for the materials and instrumentation germane to this research. A standard sample mass of 8 mg is recommended.

Shelf life and storage effects were examined, and the results showed that cables stored for six months suffered only a slight loss in OIT compared to unstored samples for all methods of storage. Sealed, refrigerated conditions are recommended for storage. Grinding just prior to OIT testing is also recommended.

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Appendix Comparison of Methods Used in the Present Research for Measuring OIT for Cable Insulation with Applicable ASTM Standards

	ASTM D 3895-94	ASTM D 4565-90a	University of Virginia
Title	*Standard Test Method for Oxidative Induction Time of Polyolefins by Differential Scanning Calorimetry,* Vol. 8.02.	*Standard Test Methods for Physical and Environmental Performance Properties of Insulations and Jackets for Telecommunications Wire and Cable,* Vol. 10.20, Section 17, "Oxygen Induction Time (Polyolefin Insulation Only."	*Reduction of Oxidation Induction Time Testing to Practice as a Life Assessment Technique for Cable Insulation,* EPRI Report, 1996, Section 8, "Development and Standardization of OIT Methodology."
Equipment	Differential Scanning Calorimetry (DSC).	DSC: "Perkin-Elmer's DSC and DuPont's Differential Thermal Analyzer with a DSC cell have been found to produce acceptable results. Equivalent equipment producing comparable results may be used.	Perkin-Elmer DSC.
Sample preparation	Compression molding. 0.250 ± 0.015 mm thick sheet. Disk 6.4 mm diameter.	Compression molding. 0.40 ± 0.04 mm thick sheet. Disk 2 is mm in diameter.	Ground to 20 mesh size in a Wiley Mill. Particle diameters from 0.85 to 1.685 mm. Recommended 20 mesh size. Effects of particle size and shape were investigated.
Sample mass	5–10 mg.	1–2 mg.	Recommended 8 mg. Effect of sample mass was investigated.
Pans	Aluminum or copper. Do not crimp or seal.	Aluminum or copper. Use 316 stainless screen (40 mesh) to cover samples in pan. Crimp the pan to hold screen.	Aluminum. Used 316 stainless screen (40 mesh) to cover samples in pan. Crimped the pan to hold screen.

Gases	Nitrogen: ultra-high purity; extra dry. Oxygen: ultra-high purity; extra dry.	Nitrogen: Commercial cylinder. Oxygen: equal to or better than 99.6% (extra-dry grade).	Nitrogen: Commercial cylinder. Oxygen: High purity.
DSC temperature	Generally 200°C. Can be varied for particular material, typically between 180 and 220°C.	No mention of DSC temperature.	200 C for most of the research.
Heating rate	20°C min.	20°C min.	20°C min.
Nitrogen flow rate	50 ± 5 mL min.	60 ± 10 mL min.	Used flow meter setting on Perkin-Elmer DSC recommended by manufacturer, setting number "15 to 16".
Oxygen flow rate	50 ± 5 mL min.	50 ± 5 mL min.	Used flow meter setting on Perkin-Elmer DSC recommended by manufacturer, setting number "15 to 16"
Added notes on gas operation	After reaching temperature, equilibrate for 5 min, then switch to oxygen. Continue isothermal operation until at least 2 min after steepest point of exotherm.	After reaching thermal equilibrium, switch to oxygen.	Began flowing oxygen to sample 4.5 min after reaching temperature. Continued isothermal operation beyond 2 min after steepest point of exotherm.
Graphical method to obtain OIT from exotherm	Instructions, with representative exotherm.	Instructions, with a representative exotherm.	Used same graphical method as in standards.
Uncertainty analysis	Precision: recommendations on repeatability and reproducibility. Bias: Stated that no standards exist.	No mention of treatment of uncertainties.	Developed an uncertainty analysis more complete than the standards.
Effect of shelf life	Not mentioned.	Not mentioned.	Evaluated effect of shelf life on OIT.

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