

UL 746B

Polymeric Materials – Long Term Property Evaluations

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UL Standard for Safety for Polymeric Materials - Long Term Property Evaluations, UL 746B

Fifth Edition, Dated August 15, 2018

SUMMARY OF TOPICS

This revision of ANSI/UL 746B dated October 3, 2022 includes the removal of dated references from ASTM Standards Referenced; 9.1.1.

Text that has been changed in any manner or impacted by UL's electronic publishing system is marked with a vertical line in the margin.

The revised requirements are substantially in accordance with Proposal(s) on this subject dated August 26, 2022.

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Fifth Edition

August 15, 2018

This ANSI/UL Standard for Safety consists of the Fifth Edition including revisions through October 3, 2022.

The most recent designation of ANSI/UL 7468 as an American National Standard (ANSI) occurred on October 3, 2022. ANSI approval for a standard does not include the Cover Page, Transmittal Pages, and Title Page.

Comments or proposals for revisions on any part of the Standard may be submitted to UL at any time. Proposals should be submitted via a Proposal Request in UL's On-Line Collaborative Standards Development System (CSDS) at https://csds.ul.com.

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INTRODUCTION

1 Scope

- 1.1 These requirements cover long-term test procedures to be used for the evaluation of materials used for parts intended for specific applications in end products.
- 1.2 Together with the Standards mentioned in Supplementary Test Procedures, Section $\underline{3}$, these investigations provide data with respect to the physical, electrical, flammability, thermal, and other properties of the materials under consideration and are intended to provide guidance for the material manufacturer, the molder, the end-product manufacturer, safety engineers, and other interested parties.

2 References

2.1 Any undated reference to a code or standard appearing in the requirements of this standard shall be interpreted as referring to the latest edition of that code or standard.

3 Supplementary Test Procedures

- 3.1 The Standard for Tests for Flammability of Plastic Materials for Parts in Devices and Appliances, UL 94, covers flammability of polymeric materials used for parts in devices and appliances. The Standard for Polymeric Materials Short Term Property Evaluations, UL 746A, contains short-term test procedures to be used for the evaluation of materials used for parts intended for specific applications in electrical end products. The Standard for Polymeric Materials Fabricated Parts, UL 746D, contains requirements for traceability and performance of parts molded and fabricated from polymeric materials.
- 3.2 Programs for the investigation of material part modifications, such as the plating of plastics or the use of flame-retardant paints, are contained in the Standard for Polymeric Materials Use in Electrical Equipment Evaluations, UL 746C.
- 3.3 Data concerning the effect of various environments and contaminants upon the properties of materials can also be obtained through standard test procedures. The more commonly used procedures are briefly described in the Standard for Polymeric Materials Short Term Evaluations, UL 746A.
- 3.4 Test procedures are provided in the Standard for Polymeric Materials Use in Electrical Equipment Evaluation, UL 746C, for the evaluation of polymeric materials in specific applications in end products. These test procedures include references to the data obtained from the standard property tests as well as other practical means of evaluation.
- 3.5 Requirements for materials that have been modified to match the requirements of a specific application, including the use of recycled and regrind materials, the use of additives and colorants, and the blending of two or more materials, are described in the Standard for Polymeric Materials Fabricated Parts, UL 746D.
- 3.6 A method and tool for the statistical analysis of long term property evaluation data and including the determination of a temperature index (TI) is described in Sections 22A 22J based on the Standard for Electrical insulating materials Thermal endurance properties Part 3: Instructions for calculating thermal endurance characteristics, IEC 60216-3.

4 Characteristics of Polymeric Materials

4.1 Polymeric materials include thermoplastic, thermosetting, and elastomeric materials. A thermoplastic material can be easily softened and resoftened by repeated heating. A thermosetting material cures by

chemical reaction and, when cured, cannot be resoftened. An elastomeric material is capable of being stretched at room temperature to at least twice its length under low stress and recovers to its original length when released from the stress.

- 4.2 Characteristics of polymeric materials that necessitate additional consideration include:
 - a) Mold stresses
 - b) Insulating quality
 - c) Resistance to ignition
 - d) Extinguishing characteristics
 - e) Production of smoke and gases
 - f) Mechanical Strength
 - g) Compatibility with solvents
 - h) Melting or distortion
 - i) Cold flow, if under stress
 - j) Fuel contribution
 - k) Dimensional stability

5 Use of Polymeric Materials

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 The rist of 5.1 The reduction to an acceptable level of the risks of electric shock, fire, and personal injury from electrical equipment depends upon the selection of materials, design, and processing of parts as well as the assembly, mounting, and relative positions of these parts.
- 5.2 The properties needed by individual parts are defined by the function or functions of the part. An enclosure, for example, must ordinarily be designed to withstand mechanical abuse. Accordingly, a material known to have substantial impact strength would normally be used although a material that has a lower impact strength, but is reinforced, might also be acceptable.
- 5.3 Electrical equipment of necessity employs many materials that usually have divergent properties. The ability to match the demands of the application with the characteristics of a material as well as the ability to compare the properties of one material with those of another can lead to an acceptable selection of materials.
- 5.4 The information gained from the data obtained from these tests can be used as an aid in the evaluation of electrical equipment using parts made of polymeric materials. Knowledge of materials can be obtained from an analysis of data from standard tests conducted on small specimens.

DETERMINATION OF THE RELATIVE THERMAL INDICES OF POLYMERIC MATERIALS

6 General

6.1 A relative thermal index of a material (RTI) is an indication of the material's ability to retain a particular property (physical, electrical, etc.) when exposed to elevated temperatures for an extended period of time. It is a measure of the material's thermal endurance. For each material, a number of relative thermal indices can be established, each index related to a specific property and a specific thickness of the material.

- 6.1A A temperature index of a material (TI), Section <u>22A</u>, is an alternative indication of the material's thermal endurance. It can as well be determined for a set of specific properties and thicknesses.
- 6.2 In determining the relative thermal index of a material, the basic concepts to be followed are stated in the Institute of Electrical and Electronics Engineers Specifications No. 1, General Principles for Temperature Limits in the Rating of Electrical Equipment; No. 98, Guide for the Preparation of Test procedures for the Thermal Evaluation of Electrical Insulating Materials; No. 101, Guide for the Statistical Analysis of Thermal Life Test Data.
- 6.3 The relative thermal index of a material is to be based upon an evaluation of long-term thermal-aging data obtained under the program described in Relative Thermal Index Based Upon Long-Term Thermal-Aging Programs, Section 8. Thermal indices on a generic basis have been established through knowledge of extensive field-service records, as outlined in Relative Thermal Index Based Upon Historical Record, Section 7. Relative thermal indices may also be established based upon a study and evaluation of the interrelationship of all of the data mentioned in Supplementary Test Procedures, Section 3, which can also be coupled with knowledge concerning the material's performance in insulating systems gained through experience or long-term aging tests.
- 6.4 A comparison of the thermal-aging characteristics of one material of proven field service at a particular temperature level with the thermal-aging characteristics of another material with no field service history provides a means for estimating the relative thermal index level at which the second material might also provide acceptable field service.
- 6.5 Another explanation of a relative thermal index is the maximum temperature below which a material maintains its characteristics over a reasonable period. This relative thermal index serves the very great need to evaluate materials that are exposed to heat sources in electrical products in which they are not used as part of an insulating system and in which they are not subjected to other major degradation influences. It is to be assumed that neither excessively long nor excessively short duty cycles are involved.
- 6.6 To be valid for use in a specific application, a relative thermal index of a material must be established by a study of the degradation rates of all properties that are relied upon in that application. As a corollary to this principle, more than one relative thermal index can be assigned to a material depending on the relative degradation rates of the properties of the material and depending on which of these properties are considered in establishing the indices.

7 Relative Thermal Index - Based Upon Historical Record

- 7.1 <u>Table 7.1</u> presents a list of materials, each of which is assigned a relative thermal index based on acceptable service experience, the chemical structure of the material, and a knowledge of the performance of the material in tests of insulating systems and electrical equipment. The assigned relative thermal index is applicable to each member of the generic material class.
- 7.2 Unless otherwise indicated in <u>Table 7.1</u>, the generic thermal index of a material is to be considered 50°C (122°F).
- 7.3 Unless otherwise indicated in $\underline{\text{Table 7.1}}$, the generic thermal index of a material is independent of thickness and pigmentation.

Table 7.1 Relative thermal indices based upon past field-test performance and chemical structure^a

Material	ISO designation	Generic thermal index,°C
Polyamide ^b	PA	65
Polycarbonate ^{b,m}	PC	80
Polycarbonate/Siloxane Copolymer ^j	PC/Siloxane	80
Polyethylene terephthalate –		
molding resin ^b	PET	75
film or sheet	PET	105
Polybutylene (polytetramethylene) terephthalate ^b	PBT	75.9
Polyphenylene Ether (including PS, PA, PP, or TPE modified) ⁱ	PPE	65 65 105 105 130 85 130 130 150
Polypropylene ^{b,g}	PP	65
Polyetherimide	PEI	105
Polyethersulfone	PES	105
Polyether Ether Ketone	PEEK	130
Polyphthalamide ^l	PPA 🗸	85
Polyphenylene Sulfide ^b	PPS	130
Polyimide film or sheet	PI	130
Molded Phenol Formaldehyde ^c	PF PF	150
Molded Melamine Formaldehyde ^{c,d} and Molded melamine formaldehyde/phenol formaldehyde ^{c,d} –	MEMF/PF Click PTFE	
specific gravity < 1.55	The state of the s	130
specific gravity ≥ 1.55	1,40	150
Polytetrafluoroethylene	PTFE	
Without inert fillers and/or reinforcements	·	180
With inert fillers and/or reinforcements		130
Polychlorotrifluoroethylene	PCTFE	150
Fluorinated ethylene propylene	FEP	150
Poly(tetrafluoroethylene, hexafluoropropylene, vinylidenefluoride) ^k	TFE/HFP/VDF	130
Ethylene/Tetrafluoroethylene	E/TFE	105
Urea Formaldehyde ^c	UF	100
Acrylonitrile – butadiene – styrene ^b	ABS	60
Silicone – molding resin ^{c,d}		150
Silicone rubber –		
molding resin	SIR	150
addition-cure, vinyl, platinum catalyzed		150
room-temperature vulcanizing, condensation or heat-cured paste	RTV	105
Ероху –		
molding resin ^{c,d}		130

Table 7.1 Continued on Next Page

Table 7.1 Continued

Material	ISO designation	Generic thermal index,°C
powder coating materials		105
casting or potting resin ^{b,h}	EP	90
Molded diallyl phthalate ^{c,d}		130
Molded unsaturated polyester ^{c,d}	UP	
alkyd (AMC), bulk (BMC), dough (DMC), sheet (SMC),		
thick (TMC), and pultrusion molding compounds		105 ^e (electrical) 130 (mec <mark>han</mark> ical)
Liquid crystalline thermotropic aromatic polyester ^f	LCP	
Ligno-cellulose laminate		60
Vulcanized fiber		90
Cold-molded phenolic, melamine or melamine-phenolic compounds ^d –		W.
specific gravity< 1.55		130
specific gravity ≥ 1.55	-OK	150
Cold-molded inorganic (hydraulic- cement, etc.) compounds	ien the full PDF	200
Integrated mica, resin-bonded –		
epoxy, alkyd or polyester binder	in	130
phenolic binder	le de la company	150
silicone binder	118	200

^a Generic thermal index is for homopolymer and for the compounding of the same type or relative resins, either grafted or ungrafted only, unless a specific copolymer or blend is indicated. In the case of alloys, the lowest generic index of any component shall be assigned to the composite. The term "grafted" means all of the monomer reacts to form a polymer, and the polymer chain forms a chemical bond. The term "ungrafted" means that the two types of polymer chains entwine with each other by mechanical blending to form a chemical composite.

^b Includes glass-fiber reinforcement and or talc, asbestos, mineral, calcium carbonate, compounding of the same type of resins, either grafted or ungrafted and other inorganic fillers.

^c Includes only compounds molded by high-temperature and high-pressure processes such as injection, compression, pultrusion, and transfer molding and match metal die molding; excludes compounds molded by open-mold or low-pressure molding processes such as hand lay-up spray-up, contact bag, filament winding, rotational molding, and powder coating (fluidized bed, electrostatic spray, hot dip, flow coating).

^d Includes materials having filler systems of fibrous (other than synthetic organic) types but excludes fiber reinforcement systems using resins that are applied in liquid form. Synthetic organic fillers are to be considered acceptable at temperatures not greater than 105°C.

^e Except 130°C generic thermal index if the material retains at least 50% of its unaged dielectric strength after a 504-hour exposure at 180°C in an air circulating oven. Specimens are to be tested in a dry, as molded, condition. Specimens that are removed from the oven are to be cooled over desiccant for at least 2 hours prior to testing.

function in Includes only wholly aromatic liquid crystalline thermotropic polyesters; wholly aromatic polyester/amides and wholly aromatic polyester/ethers; excluding amorphous, lyotropic and liquid crystalline aliphatic-aromatic polyesters which are aliphatic in the backbone chain or main chain, and substituted aromatic polyesters (except for methyl or aromatic).

g Includes polypropylene copolymers containing not more than 25% ethylene comonomer, by weight.

h Multi-part liquid epoxy materials incorporating acid anhydride or aromatic amine curing agents receive a 130°C generic thermal index.

ⁱ Includes only those polyphenylene ether materials (polystyrene, polyamide, polypropylene, or thermoplastic elastomer modified) in which the PPE component is not less than 30% of the total composition by weight and that have a Heat Deflection Temperature of at least 70°C at a load (fiber stress) of 1.80 M Pa (264 psi).

^j PC/Siloxane Copolymers in which siloxane comprises less than, or equal to, 5% of the total material composition by weight.

Table 7.1 Continued

Material				ISO	desi	gnation		Gei	neric th	ermal index,°C	
k		141	 £ 100 00			0=0() (D=					

^k Must have a minimum peak melting point of 160 °C, with less than 25% VDF monomer by weight and the remainder being fully fluorinated monomers.

¹ PPA definition according to ASTM D5336: polyphthalamide, PPA, n-a polyamide in which residues of terephthalic acid or isophthalic acid or a combination of the two comprise at least 55 molar percentage of the dicarboxylic acid portion of the repeating structural units in the polymer chain.

Additionally, this definition includes only those polyphthalamide materials that have a Glass Transition Temperature (Tg) of at least 85°C, when determined through second-heat DSC testing in accordance with the Differential Scanning Calorimetry, Section 47 of the Standard for Polymeric Materials – Short Term Property Evaluations, UL 746A.

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m Non-aromatic Polycarbonates including those based on isosorbide monomer receive a generic RTI assignment of 50°C.

8 Relative Thermal Index - Based Upon Long-Term Thermal-Aging Programs

- 8.1 The properties of a polymeric material degrade with time when exposed to various environments. A prime cause of degradation is exposure to heat.
- 8.2 The behavior of a material that is subjected to thermal aging in air cannot be assumed to be the same as its behavior under service conditions; however, a knowledge of the thermal-aging behavior of a material can be used as a basis for comparison of polymeric materials.
- 8.3 The thermal-aging characteristics of a material can be determined by measuring the changes in its properties to a predetermined level by aging test specimens at each of several elevated temperatures; plotting log of time-to the specified end point at each temperature against the reciprocal of absolute temperature; and plotting the best-fit straight line by regression analysis. The plotted line is often referred to as the life-line of a material.
- 8.4 Unless specified otherwise, a 50-percent loss of property value due to thermal degradation shall be considered as the property end point.
- 8.5 The manufacturer of the material is responsible for:
 - a) The estimation of the different applications in which the material can be used, and
 - b) The selection of the temperatures, properties, and thicknesses that are to be measured during the thermal-aging investigation.

If products of decomposition of one material are suspected of having an adverse effect on the other, for example, if two materials are not of the same generic type or if a flame retardant or other additive in one material adversely affects the other material, then they should not be aged simultaneously in the same oven. It is desirable that the oven exhaust be positively vented outside the test facility.

9 Apparatus

9.1 Ovens

9.1.1 The thermal-aging ovens that are used in the aging program shall comply with the Standard Test Methods for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation, ASTM D5374, and with the Standard Specification for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation, ASTM D5423 for Type I ovens, primarily with respect to Rate of Ventilation, Set Temperature, Temperature Variation and Thermal Lag Time.

Exception: Non air-circulating static ovens and/or forced-draft circulating-air convection ovens not capable of providing replacement of fresh air at the rate of not less than 5 changes per hour may be employed provided that:

- a) The oven is capable of maintaining the Set Temperature, Temperature Variation and Thermal Lag Time described in ASTM D5423. The Thermal Lag Time is not applicable if the oven is not subjected to frequent openings, and if the ratio of oven aging time to open-oven time is large.
- b) The products of the material decomposition are not expected to further degrade the polymer in other words, shall not be autocatalytic, and
- c) A control material of known performance is aged in the same ovens and for the same time duration as the candidate materials.
- 9.1.2 The oven temperature control is to be capable of long-term operation. It is desirable that the oven be provided with a timer and also with an oven-temperature cut-off to prevent loss of specimens, loss of data, or loss of test continuity. Ordinarily, at least four ovens of applicable capacity are needed to contain the aging specimens; however, two ovens can be effectively used by completing the work at the two higher thermal-aging temperatures first and then switching the oven settings to the two lower thermal-aging temperatures.
- 9.1.3 Temperatures for heat aging are to be accurately controlled and monitored. At the start of the program, oven temperatures are to be checked frequently. The use of several thermocouple locations to check variations throughout the ovens is required. As the test progresses, monitoring can be done less frequently.

10 Scope of Test Programs

10.1 Selection of test properties

- 10.1.1 The specific properties to be evaluated in the thermal-aging program shall be determined.
- 10.1.2 The contemplated applications of the material (as intended by the manufacturer of the material), the flammability characteristics, and the physical and electrical properties that the material needs to have for these applications are to be considered.
- 10.1.3 <u>Table 10.1</u> provides a list of properties that can serve as an aid in the determination of the properties to fall within the scope of the investigation. The properties are to be as nearly as possible representative of the properties required for the end application.

Table 10.1
List of properties and test methods

Property ^a	Test Method
Mechanical Properties	
Maximum Tensile Stress or Flexural Strength	UL 746A
Tensile Impact, Izod Impact, or Charpy Impact ^b	UL 746A
Electrical Properties	
Dielectric Strength	UL 746A
Flammability Properties	

Table 10.1 Continued

Property ^a	Test Method
Vertical Burning	UL 94

^a The list of properties given in this table is not complete. Other properties that are critical in a particular end-use application are to be included in the program.

10.1.4 The results of Tensile, Charpy or Izod Impact testing of standard specimens in either the nominal 3 mm or 4 mm thickness, as appropriate for the specified test method, can be considered representative of the testing of reduced thicknesses provided such reduced thicknesses have been evaluated for non-impact mechanical properties. The assigned relative thermal indices for impact properties in the reduced thicknesses shall be lowered by an offset equal to the corresponding lower offset, if any, of the relative thermal indices of the non-impact properties at the reduced thicknesses. Table 10.2 illustrates a hypothetical example of this offset. It is appropriate to consider temperature interval classification described in 23.1 while assigning the RTI-impact values at <3 mm thickness.

Table 10.2
Example of applying offset principle to assigning impact ratings

	Measured RTI		Assigned RTI considering the temperature interval classification			
Min. thick. (mm)	Elec	Imp	Str	Elec	Imp	Str
0.75 ^c	245ª	169 ^b	231 ^a	240 ^d	160 ^d	220 ^d
1.5	245	171 ^b	233 ^a	240 ^d	170 ^d	220 ^d
3.0	245	183ª	245 ^a	240 ^d	180 ^d	240 ^d

^a Measured Thermal Indices assigned based on actual testing at thicknesses.

10.1.5 The term Measured Relative Thermal Index (or Indices) represents the relative thermal index (or indices) of the material under investigation as determined by use of the relevant time-temperature plot without adjustments based on the application of the temperature interval classifications defined in 23.1. Table 10.2 illustrates a hypothetical example of this offset principle.

11 Property-Evaluation Tests

11.1 General

- 11.1.1 <u>Table 10.1</u> provides a list of test specifications that generally are used for property-evaluation tests. Other tests can be used if found to be acceptable for the application.
- 11.1.2 The tests are to monitor the performance level for each property as the accelerated aging of the material progresses.

^b For unaged materials that do not exhibit break upon impact testing, the RTI Mechanical Impact can not be determined and therefore the RTI Mechanical Impact is not applicable (N/A).

b Measured Thermal Indices assigned based on the results of testing the 3.0 mm or 4.0 mm thickness, reduced by the corresponding offsets of 245°C − 233°C = 12°C and 245°C − 231°C = 14°C at 1.5 and 0.75 mm, respectively.

^c Offset principle for impact ratings also applies to minimum thicknesses less than 0.75 mm provided that they have been tested to <u>Table 10.2</u> requirements.

^d Relative Thermal Indices assigned for a given grade based on the Measured Thermal Indices at different thicknesses considering the temperature interval classification mentioned in 23.1.

11.1.3 The tests selected are to simulate as closely as possible the field-service conditions of the contemplated-use application. Some test methods can be used for only certain forms of polymerics (for example, film or sheet materials).

11.2 Choice of end-point

- 11.2.1 The Institute of Electrical and Electronic Engineers (IEEE) standards do not specify the method of determining end of life, although several alternatives are presented. Fixed property level and percent-of-unaged property level are two of these that appear to have the most significance in relation to end-use applications. Product design normally involves the factor-of-safety approach. Therefore, the relative thermal index developed by this standard is based on the assumption that a factor of safety exists in the applicable physical and electrical property requirements. It is not expected that a 50-percent loss of property due to thermal degradation results in premature risk of electric shock, fire, or personal injury. The considerations have led to the decision to report the end point at each aging temperature as the time at which a property value has decreased to 50 percent of its unaged level where quantifiative evaluation test methods are available.
- 11.2.2 In certain applications, the reduction to 50 percent of the initial property value may not represent the stresses encountered in actual service. A fixed property level may be used in applications where the anticipated service stress can be defined and where consideration has been given to the expected duty cycle, degree of deterioration over the useful product life and an acceptable factor-of-safety. As an alternative, the relative thermal capability may be determined for each application using the concepts described in the Standard for Polymeric Materials Use in Electrical Equipment Evaluations, UL 746C.

12 Sampling Programs

- 12.1 Two sampling techniques are available for conducting a long-term thermal aging investigation: the Fixed Temperature Method in Sections $\underline{13} \underline{21}$ and the Fixed Time Method in Section $\underline{22}$. Both methods will provide the time-temperature-property values needed to establish the Thermal Index Rating and assign a thermal class rating. The primary difference between the methods is in the sampling technique employed. Since both methods rely upon a data analysis of the degradation of samples at various temperatures and using specific time intervals, the results of the tests would be expected to be similar regardless of which of the two methods is selected.
- 12.2 Both the control and the candidate materials shall be evaluated using the same sampling method (Fixed Temperature or Fixed Time).
- 12.3 The test specimens are to be the same size and shape for both test methods.
- 12.4 All material properties (i.e., electrical, mechanical, and flammability) can be evaluated using either method.
- 12.5 All initial (unaged) and aged specimens are to be tested using the same test method for each property.

13 Selection of Oven Temperatures

13.1 At least four oven temperatures are to be selected. The lowest oven temperature (T4) selected is to produce an anticipated end point of the material's property at this temperature in not less than 5,000 hours and shall not be lower than the Relative Thermal Index (RTI) ultimately assigned. The highest oven temperature (T1) selected is to produce an anticipated end point of the material's property at this temperature in not less than 500 hours. The minimum aging time criterion is applicable for each primary property evaluated. See <u>Table 13.1</u>.

- 13.2 Degradation is a function of the aging characteristics of the particular polymer. Specific aging temperatures cannot therefore be recommended since the test temperatures can differ for each material tested.
- 13.3 Short-time screening tests at various temperatures can be used to estimate the anticipated end-of-life.
- 13.4 If degradation cannot be accelerated because of transition points or threshold temperatures, and in consideration of the need for a spread between aging temperatures, it might be necessary to extend the low-temperature test (t4 in <u>Table 13.1</u>) to well beyond the usual 5000-hour minimum value to obtain significant data. However, if the level of the evaluated property is still above the end point criteria at 10,000-hours of aging this is considered sufficient evidence for its thermal performance. One of the options shown in <u>Table 13.2</u> may be followed for analysis and evaluation.

Table 13.1 Selection of oven temperatures

Test temperature (°C)	t1 (highest)	t2	t3	t4 (lowest)
End Point (Hours)	500 min.	1,500 approx.	3,000 approx.	Shall not be lower than the expected/assigned RTI of Candidate 5,000 min.
Cycle Period ^a Days	3	7	14	28
^a See <u>15.2</u> .		0	•	•

Table 13.2 Options for data analysis

Option	Analysis method
3 – 4 endpoints	a) Use less than 50-percent loss of property as criteria.
COM;	b) t1 to t3/t4 to be determined at the highest 30 to 50-percent loss of property, that is in compliance with the t1-criteria in Table 13.1.
ORM, ON	c) Same method shall be applied to the control to determine the correlation time.
70/x	d) Candidate RTI to be assigned at the control's correlation time.
2 – 3 endpoints	a) Use t1 to t2/t3 and maximum aging time at t3/t4 respectively.
	b) This method should not be used for the control.
	c) Candidate RTI to be assigned at the control's correlation time.
1 end point: (applicable for an exceptionally durable material that shows 1 end point at 10,000 hours of aging	a) Identify the maximum temperature (t °C) that does not show F50 point at the end of 10,000 hours aging.
	b) Use the above time/temperature value to draw an Arrhenius line with slope E/R = 4200.
	c) If the % retention at 10000 hours is > 80%, then the RTI rating shall be based on 20000 hours correlation time.
	d) If the % retention at 10000 hours is between 60% – 80%, then the RTI rating shall be based on 40000 hours correlation time.

13.5 The spread between aging temperatures is to be enough to overcome the small errors in measuring and controlling temperatures, generally at least 10°C (18°F).

13.6 The reason for these test-temperature limitations is to provide accurate data so that extrapolation to determine the acceptable operating temperature for life can be reasonably predicted.

14 Selection of Control Material

- 14.1 A control material is to be selected and tested in the thermal-aging test program in the same manner as the material under investigation.
- 14.2 The control material is to be a material that has an established relative thermal index. Preferably, the material is to be one with a record of good field service at its rated temperature. If possible it is to be of the same generic type as the candidate material, is to be tested in the same thickness, and is to have a relative thermal index as close as possible to that expected for the candidate material.
- 14.3 More than one control material may be tested to insure comparable performance to the candidate, but only one control will be considered in establishing the candidate's RTI for all properties.
- 14.4 The control shall be tested at the same time as the candidate and conditioned in the same ovens except where the performance ranges of candidate and control are sufficiently different to necessitate different ranges of aging temperatures, or where special contamination problems have been demonstrated. If different ovens are necessitated because of different ranges of aging temperatures, then at least two of the four aging temperatures shall overlap and the same ovens, containing both control and candidate test specimens, shall be used at these overlapping temperatures. The property end points for both the candidate and control material test specimen conditioned in these overlapping ovens shall comply with the minimum aging times indicated in Table 13.1.

15 Specimens

- 15.1 The physical dimensions of the test specimens are given in the test specifications referred to in the Standard for Polymeric Materials Short Term Property Evaluations, UL 746A. Normally, 5 specimens constitute a set; however, a greater number might be necessary if the property under evaluation exhibits scattered results. Refer to Aging, Specimen, and Check-Test Schedules, Section 21 for typical sample requirements.
- 15.2 For each oven temperature, there is to be an assigned cycle period. Usually the cycle period for the highest temperature is to be 3 days, for the next lower temperature 7 days, for the next lower temperature 14 days, and for the lowest temperature 28 days. Refer to <u>Table 21.2</u>.

16 Thermal Aging

- 16.1 To obtain a measurement of each of the properties at the end of each successive cycle period for each oven-aging temperature, it might require an extremely large number of specimens inasmuch as the material generally survives more than 10 cycles of the test program.
- 16.2 To conserve on the total number of specimens required, to reduce the frequency of making the measurements, and also to develop data near the time of 50-percent reduction of the initial property value, the procedure in $\underline{16.4} \underline{16.6}$ can be followed.
- 16.3 Specimens of unaged set(s) are to be pre-conditioned for 48 ±8 hours at the lowest aging temperature used at the beginning of the program for the corresponding property evaluated to eliminate any short-term thermal effects.

NOTE: For example, if the T4 oven temperature for Electrical Property and Impact property at the beginning of the program is determined to be 140 $^{\circ}$ C (284 $^{\circ}$ F) and 120 $^{\circ}$ C (248 $^{\circ}$ F) respectively, then the unaged set for Electrical and Impact Property evaluation shall be pre-conditioned at 140 $^{\circ}$ C and 120 $^{\circ}$ C respectively.

- 16.4 Initially for each temperature, 5 sets of specimens are to be placed in the oven. At the end of the first, second, and third cycles an additional set is to be added.
- 16.5 At the end of the third cycle, some of the original test specimens are to be removed from the oven and subjected to the applicable tests. Assuming that the average property value of these specimens is greater than the end point, the property test is to be repeated on conditioned test specimens every successive third cycle until the results of the property testing are equal to or less than the end point. In common practice, these specimens are selected from the test specimens initially put in the oven.
- 16.6 When the results of the property testing are equal to or less than the end point , the sets of specimens that were placed in the oven at delayed times are to be removed from the oven and tested. Their performance analysis can result in a more accurate determination of the time to reach the property end point . If the property end point is not obtained at the time that all of the original specimens have been tested, the delayed specimens can be removed from the oven at various times in the test program in order to extend the aging time. This general procedure is to be followed for all tests that involve the determination of the property end point of the test specimens. Table 16.1 summarizes this approach. A typical data sheet that can be used to record the summary of the thermal-aging testing is shown in Figure 16.1.
- 16.7 Using this technique, a numerical value for each property is to be obtained at the end of each cycle. It is possible to plot a curve showing the relationship between the values of each property and time at each of the 4 aging temperatures. This end point data can then be used to determine a relative thermal index.
- 16.8 At least two additional data points should be obtained after reaching the property end point to confirm the end-of-life value. These data points should be as close as possible to the property end point to provide a more accurate calculation of the end-of-life value.
- 16.9 The determination of the properties may require calculations that include the dimensions of the specimens. In such cases, the dimensions of the specimens prior to oven conditioning should be recorded and used in the property calculation.

Exception: For physical properties, if the dimensions of the specimens significantly change as a result of the oven conditioning, the dimensions that result in the lower physical property value should be reported and used in the property calculation.

Table 16.1 Delayed set test procedure primary properties

End of cycle number	Sets put in oven	Sets tested
0	B, C, D, E, F	A (unaged)
1	G ^a	-
2	H ^a	-
3	Į ^a	B ^b
4, 5, 6	_	_
7	_	C _p
8, 9, 10	_	-
11	_	D ^b
12, 13, 14	_	=0/L
15	_	ED -
16, 17, 18	_	1 1 1 1 1 1 1 1 1 1
19	_	OF JIL OF F ^b G G -
20	_	- · · · · · · · · · · · · · · · · · · ·
21	_	G
22	- 4	_
23	- OX	н
24		_
25	- "	I

^a Sets G, H, and I are to be put in the oven one or more days later than the end of cycles 1, 2, and 3, respectively. This procedure gives time for further conditioning of sets tested resulting in a decision for removal of the delayed sets.

The end of 19 cycles, sets G, H, and I are continued to the chick to t

^b Should the property end point be reached at the end of 3, 7, 11, 15, or 19 cycles, sets H and I are to be removed from the oven and tested in order to more precisely determine the property end point time. If the property end point is not reached by the end of 19 cycles, sets G, H, and I are to be tested as shown.

Figure 16.1
Thermal aging data summary (Destructive testing)

Manufacturer Material				
Sample	°C; HRS/CYCLE	Oven Temp°C; Sample Thickness Property		
		Averaged Test Value		Averaged Test Value
Cycle Number	Elapsed Hours	(Units)	Elapsed Hours	(Units)
0	0		0	
1				<u> </u>
2				2011
3			^	`V
4			.6	•
5			11	
6			N.	
7			ζ.	
8			0,	
9			OX	
10			Q v	
11		(1)		
12		Q ₁		
13		ille		
14		N		
15		jie		
16		×O		
17		+		
18	il's)		
19	,0			
20	W.			
21	60			
22	<u>, 0</u>	_		

17 End-of-Life

17.1 Primary properties

17.1.1 When there is no information as to which of the properties (flammability, dielectric strength, flexural strength, etc.) might be the first to degrade to an unacceptable value, complete testing is to be generally carried out for each property. However, where specific properties are known to degrade more rapidly, and the relative thermal index of the material is to be based on these properties, the other properties in the program are to be measured only at the end-point of the property that is tested full scale. The properties that are monitored throughout the program are to be referred to as primary properties.

17.2 Secondary properties

17.2.1 The properties that are to be measured only initially are to be referred to as secondary properties after the property end point of the prime property occurs. If the secondary-property measurements indicate the material has passed through the end point of the secondary property, retesting – with check tests

throughout aging – is then required to establish the life-temperature relationships. Delayed sets of specimens can effectively be used in this case. See Table 17.1.

Table 17.1				
Delayed set test	procedure – secondar	y properties		

End of cycle number	Sets put in oven	Sets tested
0	N	M (unaged)
1	0	-
2	Р	-
3	Q	-
4	R	- 07
5, 6, 7 etc.	_	AOV

^a All sets are to be removed from the oven and tested at the time that the earliest primary property passes through the 50-percent point as determined by the method shown in <u>Table 16.1</u>. If set N shows that it does not pass through the 50-percent point, the remaining sets need not be tested. If set N passes through the 50-percent point, then sets O, P, Q, and R are to be tested in turn. These sets are not aged as long as those initially put in the oven.

18 Proof Testing

18.1 In some cases, to keep the number of specimens in the oven to a minimum, proof testing can be employed. In this case, the property is not to be measured in an absolute manner on aged specimens. Instead, the numerical value of the property is to be determined on unaged specimens to establish a reference value. At the end of each cycle during the aging-test program (see Table 16.1), all test specimens (usually 10) are to be subjected to a property stress at a level of 50 percent of the initial property value. Specimens that do not have the ability to comply with this property stress are to be removed from the test program and the length of time each specimen was in the oven is to be noted. The end-of-life is to be assumed as having occurred half way through the cycle preceding removal of the specimen from the oven. Specimens that have the ability to comply with the property stress are to be returned to the oven for further aging, and the property stress is to be repeated at the end of the following cycle. This procedure is to be continued until the property end point for all specimens is obtained. The average log life is to be determined and used to establish a relative thermal index. This type of proof testing usually is to be employed when dielectric strength is the property to be evaluated. In this case, only a single end point can be determined, and this is usually 50 percent of the initial value of the property.

Exception No. 1: It has been observed from empirical data, that the logarithm of time to degrade to 50 percent of the initial property level is generally distributed normally at any given temperature. The probit method of analysis described in the National Institute of Standards and Technology Handbook 91 entitled Experimental Statistics, may be employed to estimate the log average life, provided that at least half of the samples have reached the property end point at that test temperature.

Exception No. 2: For polypropylene, observation of crazing on 10 percent of the total surface area of the test specimen, rather than 50 percent retention of the initial property value, is to be used in determining the property end point time.

19 Analysis and Evaluation

- 19.1 After accumulating the data, it is necessary to evaluate the insulating material in terms of operating temperature and life expectancy. Also, it is important to provide a clear statement of the accuracy and uniformity of these results so that the degree of reliance can be determined.
- 19.2 When destructive testing is employed, it is first necessary to determine the aging time at which the property level decreases to the property end point at the accelerated-aging temperature. The degradation

mechanism is usually a complex combination of effects due to chain scission, oxidation, change in crystalinity, formation of a dense cross-linked skin, etc., and the time-temperature relationship may not accurately be defined in terms of a continuous simple relationship. It may be possible to generate a simpler relationship by transforming the graph of property versus time at the different aging temperatures into discrete strength lines by use of applicable functions of time, u = f(t) or property, v = f(p).

19.3 If an acceptable amount of data can be obtained around or near the property end point, a third-order polynomial equation is useful to interpolate most of the data that is encountered. This method generally is not to be used for extrapolation to the property end point. The equation has the form:

$$y = a_0 + a_1 t + a_2 t^2 + a_3 t^3$$

in which:

y is a measure of the attribute (property level), and

t is time expressed in hours.

Other relationships may be employed in place of the best-fit third-order polynomial if it can be shown that a better portrayal of the data set is achieved.

19.4 The polynomial constants may be solved by using the following matrix equation:

$$\begin{bmatrix} \Sigma y \\ \Sigma ty \\ \Sigma t^2 \end{bmatrix} = \begin{bmatrix} n\Sigma t \Sigma t^2 \Sigma t^3 \\ \Sigma t \Sigma t^2 \Sigma t^3 \Sigma t^4 \\ \Sigma t^2 \Sigma t^3 \Sigma t^4 \Sigma t^5 \\ \Sigma t^3 \Sigma t^4 \Sigma t^5 \Sigma t^6 \end{bmatrix} \cdot \begin{bmatrix} \alpha_0 \\ \alpha_1 \\ \alpha_2 \\ \alpha_3 \end{bmatrix}$$
Say 15

In the equation, n is the number of data points used in the calculations and all summations are from 1 to n. This represents four equations with four unknowns, and these can be used to solve for the coefficients a_0 , a_1 , a_2 , and a_3 in terms of the known sums determined from the data points. Usually, at least five data points are required to establish a useful relationship.

19.5 For the purpose of illustration, consider the following data set:

e (elapsed hours)	Tensile strength (MPa)		
0	84.5	I	
504	92.6	I	
1478	53.8	1	
1915	37.2	}	Data Points used in the example calculation
1948	35.9	1	
1982	39.9	1	
2016	36.4	1	

A judgment was made to eliminate the 0-hours, 84.5-megapascal data point from the matrix equation due to its distance away from the property end point. The matrix of $\underline{19.4}$ is generated in a manner that is typified by the following:

$$\Sigma t = 504 + 1478 + 1915 + 1948 + 1982 + 2016$$

$$\Sigma t^{2} = (504)^{2} + (1478)^{2} + (1915)^{2} + (1948)^{2} + (1982)^{2} + (2016)^{2}$$

$$\Sigma t^{3}y = (504)^{3}92.6 + (1478)^{3}53.8 + (1915)^{3}37.2 + (1948)^{3}35.9 + (1982)^{3}39.9 + (2016)^{3}36.4$$

When the simultaneous equations derived from the matrix equation are solved, the following polynomial equation is obtained to represent the data:

$$y = 43.4075 + (1.813 \times 10^{-1}) t - (1.9084 \times 10^{-4}) t^2 + (4.936 \times 10^{-8}) t^3$$

At the property end point, y = 1/2 (initial property level). In the example, this corresponds to y = 42.25 megapascals. The value of t when y = 42.25 megapascals may be determined by iteration, using computer techniques. A calculated time of 1707 hours was determined using the best-fit cubic polynomial. An alternative is to express the equation coefficients as a function of percent property retention (z) versus time (t). For this alternative, the cubic equation is expressed as:

$$z = 51.436 + (2.2576 \times 10^{-1}) t - (2.2576 \times 10^{-4}) t^2 + (5.8390 \times 10^{-8}) t^3$$

The value of time corresponding to z = 50 percent initial property value may be calculated as 1707 hours.

- 19.6 The life expectancy is to be considered a function of temperature. The Arrhenius equation describing the temperature dependence of the velocity coefficient of chemical reactions can be used to approximate the relationship between material life and temperature. This equation, as applied in this case, indicates that the logarithm of material life is a linear function of the reciprocal of the absolute temperature. The best fit of the slope and intercept of the straight line that relates the logarithm of material life to the reciprocal temperature is to be determined by the least-squares method of linear regression analysis.
- 19.7 The Arrhenius equation for reaction rate is given by k=Ae to the power (-E/RT) in which k is the specific reaction rate, E is the activation energy (relatively constant for a small temperature change), R is the gas constant, T is the absolute temperature, A is the frequency factor (constant), and e is 2.718284.

19.8 The Arrhenius equation can be simplified by taking natural logarithms in the following form:

$$\log_e k = \log_e A - \frac{E}{RT}$$

letting $Y = log_e k$, $a = log_e A$, b = -E/R, and X = 1/T, we then have Y = a + bx. This relates the two variables Y and X in the form of a linear equation, assuming a and b are constants.

For RTI analysis purpose, log_e could be replaced by log_{10} . If the above equation is divided throughout by "ln(10)", the same relationship Y= a + bx is obtained as depicted below:

$$\log_{10} k = \frac{\log_e k}{\log_e 10} = \frac{\log_e A}{\log_e 10} + \left(\frac{-E}{\log_e 10 * R}\right) * \frac{1}{T}$$

Y=a + bx

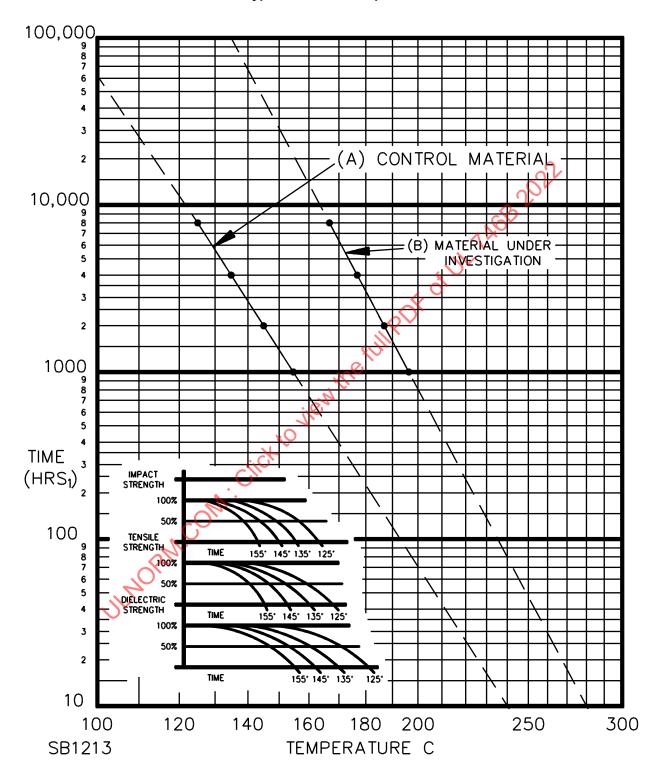
This reduces to a linear plot between 1/T - Reciprocal Temperature and log k - Specific Reaction rate.

19.9 The evaluation of the insulation is completed by the regression analysis. This method of analysis is concerned with the study of the relationship between two or more variables. In this instance, a study is to be made of the relationship between material property life and operating conditions. Property life is denoted as the dependent variable represented by the letter Y, and the operating condition as the independent variable, represented by the letter X. Thus, the regression analysis becomes a study of Y (log of specific reaction rate) as a function of X (reciprocal of operating temperature).

19.10 After the Arrhenius equations for both the candidate material and the control material are determined and plotted, a comparison is to be made to establish a relative thermal index of the candidate material.

19.11 The insert in Figure 19.1 illustrates the curve obtained as the result of aging the material under investigation at four elevated temperatures. In the example, the properties of impact strength, tensile strength, and dielectric strength are investigated. At each temperature, the first property to reduce to 50 percent of its unaged property value is impact strength. The time to reach 50 percent at each temperature for this property is then to be used to construct the time-temperature plot shown in curve B.

Figure 19.1
Plot of typical time and temperature data



- 19.12 Curve A represents the plot of a control material having a relative thermal index of 100°C (212°F), based either on previously accumulated long-time data or on the knowledge of a long, known service experience in general-use applications. This known material shows a correlation factor in this example of 60,000 hours when tested in the manner described in this program.
- 19.13 The time-temperature plot of the material under investigation crosses the 60,000-hour line at a temperature of 140°C (284°F). Therefore the material can reasonably be expected to be as useful at a temperature of 140°C (284°F) as the control material is at 100°C (212°F).
- 19.14 Since the correlation time of the Control material at which the RTI of the Candidate material is determined can vary for different material types and for different properties, the criteria mentioned in Table 19.1 shall be used to assign RTI ratings for the candidate material.

Table 19.1 Criteria for assigning candidate RTIs based on control correlation time

Control correlation time (hours)	Candidate RTI assigned at			
< 5000	5000 hours			
5000 – 60000	Corresponding Correlation Time			
> 60000	60000			
No Control (only candidate)	60000 hours (or 20000 hours for TI) ^a			
^a Requires data linearity validation according to Electrical insulating materials. Thermal endurance properties – Part 3:				

- 19.15 In the absence of comparison data for a control material, it might be difficult to correlate the longtime-endurance program with actual service conditions. There is some evidence to show that an arbitrary life of 60,000 hours under this long-time program can be assumed when determining a relative thermal index. Examination of correlation factors from prior RTI determinations indicate 60,000 hours to be a reasonable upper bound on correlation time. In place of applicable control data, an extrapolated life of 60,000 hours or an extrapolated life of 20,000 hours (applicable only to the method for Electrical insulating materials - Thermal endurance properties - Part 3: Instructions for calculating thermal endurance characteristics, IEC 60216-3) is to be used to assign the relative thermal index (RTI) or thermal index (TI) respectively. In cases where the correlation time for the control material is higher than 60,000 hours, an extrapolated life of 60,000 hours is to be used to assign the relative thermal index.
- 19.16 In considering the usefulness of the relative thermal index in the example given in Figure 19.1, consideration is to be given to the properties that are evaluated in the program. If the properties being stressed in the end product are also considered in arriving at the general-use thermal index, the relative thermal index resulting from this analysis is valid and can be used in the evaluation of the material in the end product. If the property being stressed in the end product is not evaluated in the long-term-aging program, the relative thermal index might not be applicable to the use of the material in that particular application.
- 19.17 In considering the example shown in Figure 19.1, it is possible that more than one temperature rating can result from analysis of the data accumulated during the long-time investigation. In the example described in 19.11 the most critical property being investigated is impact strength and the general-use relative thermal index of 140°C (284°F) is applicable to all applications involving all of the properties investigated, including impact strength. However, there can be applications of this material in which impact strength is not a critical property, such as in an application in which the material is shielded from mechanical abuse as is the case for some insulating materials, terminal boards, wire connectors, etc. In that event, a time-temperature plot could be made for the unknown material considering all properties except impact strength. In such an example, it might be possible to have a relative thermal index of, say

155°C (311°F), for applications in which impact strength is not a critical property and 140°C (284°F) for applications in which impact strength is required.

19.18 Care is to be exercised in the use of any general-use relative thermal index achieved by the method of analysis described in this standard. If it is felt that the end-product application of the material involves unusual service conditions, the acceptability of the material at the relative thermal index is judged by this method is to be reviewed. If service conditions associated with an end-product application are less severe than those considered in arriving at the relative thermal index, higher operating temperatures may be acceptable.

20 Related Material - Coverage of Variations in Material Composition

20.1 General

- 20.1.1 Commercially available brands of insulating materials are usually obtainable in different molecular weights and colors, and with differing types and quantities of fillers and additives. A separate analysis of each of these variations is not necessary to an evaluation in a thermal-endurance program.
- 20.1.2 The least favorable performance of the unfilled and maximum-level filled or reinforced material shall be considered representative of intermediate levels of filler or reinforcement without additional testing.

20.2 Thermoplastic materials

- 20.2.1 Thermoplastic materials that are related to others in the program can, in accordance with $\underline{20.2.2} \underline{20.2.15}$, and Table 9.1 and Table 9.2 of UL 746A, may be evaluated using abbreviated test programs. These abbreviated programs apply specifically to families of thermoplastic materials in which each of the related materials (candidate) is intended to have properties that are similar to or differ slightly from the basic material (control) and are assigned a same or different compound designation.
- 20.2.2 In cases where the limits in Table 9.1 of UL 746A are exceeded, and where the polymer variation is not expected to affect the material's thermal endurance characteristics, testing will include one or two temperature aging (UL 746B) using the unaltered basic material as the control reference. Both the impact and non-impact mechanical properties tested in the nominal 3 mm thickness can be considered representative of other properties and thicknesses, however, if a lowering of the non-impact mechanical index is indicated, then the electrical index not tested will be automatically lowered by the same amount and materials may need to be checked after additional aging for retention of flame retardancy.
- 20.2.3 Reference materials to be considered as the unaltered basic material for application of the limits in Table 9.1 of UL 746A, and for use as a control in any required tests, shall be a material that has actually been subjected to thermal aging tests and not a material with an assigned temperature index based solely on a previous application of this analysis.
- 20.2.4 If testing of a related material is not indicated in Table 9.1 of UL 746A, the material can be assigned the same temperature rating as the original material.
- 20.2.5 A comparison of the results of aging at one temperature (neither the highest nor the lowest used in the investigation of the basic material) with the life-line (Arrhenius curve) of the basic material is to be conducted, assuming parallel performance and extrapolated to the life value corresponding to the relative thermal index of the base material. If the difference between the extrapolated life of both materials is within 5°C (9°F), then the related material is to be assigned the same relative thermal index as that determined for the basic material. If the difference between the extrapolated lives of both materials is not within 5°C (9°F), the related material cannot be assigned a relative thermal index unless the additional aging described in 20.2.11 is conducted. See 20.2.15 for an illustrative example.

- 20.2.6 As an alternative to the Long Term Thermal Aging test described in 20.2.5, the following equivalent one temperature and single-point aging test with supporting technical information and analytical tests can also be conducted.
- 20.2.7 Supporting technical information by the manufacturer on the known formulation (the control) and the known formulation with ingredient variations (the candidate) shall be validated to obtain an indication that the variation will not lead to deterioration in long-term heat aging performance in the candidate material. Technical information such as chemical difference of the variation, molecular weight of polymer, melt viscosity of compound, crystallinity of polymer, or other thermal or oxidative characteristics before and after thermal stress etc. can be used as appropriate for the study affecting the aging performance.
- 20.2.8 Analytical tests such as Dynamic Mechanical Analysis (DMA), Thermogravimetric Analysis (TGA in air and N_2), Differential Scanning Calorimetry (DSC) and Pressure DSC (PDSC) shall be performed as appropriate to validate the technical information described in 20.2.7.
- 20.2.9 One-temperature and single-point aging test shall be conducted to determine percent property retention at 2000 hours on the control and the candidate formulation shall be performed. The aging temperature is to be chosen from the historical aging data of the control material such that the absolute value of percent property retention for the control material at the aging temperature would lie between 35 75 percent at about 2000 hours.

Exception: If the historical aging data of the control material shows percent property retention more than 75 percent or less than 35 percent at 2000 hours at a specific temperature, closest time to 2000 hours may be chosen to meet the absolute value of percent retention described in 20.2.9.

20.2.10 If a Two-Sample T-Test shows that the mean of the candidate's percent retention value is comparable or greater than the mean of the control's percent retention values, the same Relative Thermal Indices (RTI's) as the control will be assigned to the candidate independent of the p-value results.

Percent retention values are considered comparable if:

- a) The mean of the candidate's percent retention values is within \pm 5% of the mean of the control's percent retention values or
- b) A Two-Sample, 2-tailed and homoscedastic (equal variance) T-Test of the percent retention property values between the candidate and control formulations (using about 30 data points) at 2000-hour time interval is found to be statistically not significant (p-value > 0.05, at 95 percent confidence).
- 20.2.11 If comparison of the results of aging at the two mid-temperatures, used in the investigation, having the same slope as the basic material but displaced so as to have the best fit with the two new points, extrapolates to within 5°C (9°F) of the relative thermal index of basic material, the related material is to be assigned the same relative thermal index as that determined for the basic material. See 20.2.15 for an illustrative example.
- 20.2.12 If the extrapolated temperature of the related material exceeds the basic material's relative thermal index, the related material is to be assigned the same relative thermal index as that determined for the basic material.
- 20.2.13 If the extrapolated temperature of the related material is less than the basic material's relative thermal index by 5 to 10°C, the related material is to be assigned a relative thermal index value 10°C lower than that determined for the basic material. In the event that the extrapolated temperature result is lower by more than 10°C (18°F) of the basic material's relative thermal index, the related material can be assigned a relative thermal index only on the basis of an aging program at four temperatures.

20.2.14 The following data on a base material compared to data obtained on a related material aged and tested under the same procedure and condition is intended as an illustration:

Material life
Time (hours) to reach the property end point

Temperature, °C	Base material	Related material
200	1200	
190	1824	1150
180	3288	_
170	5232	_

Using the procedure in Analysis and Evaluation, Section 19, linear regression analysis on the base material's data results in the relationship:

$$\log_{10}(\text{life}) = \frac{4559.5739}{\circ C + 273.16} - 6.5641$$

A relative thermal index of 125°C is assigned to the base material, which corresponds to a 77,228 hour correlation time (life).

It is to be assumed that the slope of the related material is identical to the slope of the base material, and that the equations differ only in the value of the ordinate intercept. The equation for the related material can be found by substituting the known data point as follows:

$$\log_{10}(1150) = \frac{4559.5739}{190 + 273.16} + A$$

Hence, the relationship between time and temperature for the related material is given by:

$$\log_{10}(\text{life}) = \frac{4559.5739}{^{\circ}C + 273.16} - 6.7838$$

At the base material correlation time, the related material's relative thermal index is given by:

$$\log_{10}(77,228) = \frac{4559.5739}{^{\circ}C + 273.16} - 6.7838$$

which can be calculated as 117.5°C. This value is not within the 5°C differential indicated in 20.2.5 and the related material is not eligible for a relative thermal index unless additional tests are conducted.

20.2.15 Continuing the example in 20.2.14, assume that the manufacturer generates additional data at 180°C (356°F) that results in a material life of 2200 hours – that is,

$$T_1 = 463.16K (190 \,^{\circ}\text{C}) \,\text{Life}_1 = 1150 \,\text{hours}$$

$$T_2 = 453.16K (180 \,^{\circ}\text{C}) \,\text{Life}_2 = 2200 \,\text{hours}$$

This data can be expressed as a single arithmetic mean value as:

$$\overline{X} = \frac{(X_1 + X_2)}{2} \qquad \overline{Y} = \frac{(Y_1 + Y_2)}{2}$$

$$\overline{X} = \frac{\frac{1}{T_1} + \frac{1}{T_2}}{2} \qquad \overline{Y} = \frac{\log_{10}(\text{life}_1) + \log_{10}(\text{life}_2)}{2}$$

$$\overline{X} = \frac{T_1 + T_2}{2T_1 T_2} \qquad \overline{Y} = \frac{\log_{10}(\text{life}_1 \cdot \text{life}_2)}{2}$$

$$\overline{X} = \frac{1}{458.1054} \qquad \overline{Y} = 3.2016$$

The equation for the related material is to be found by substituting the mean data as follows:

$$3.2016 = \frac{4559.5739}{458.1054} + A_{1}$$
or
$$A_{1} = -6.7515$$

Hence, the between time and temperature relationship for the related material is given by:

$$\log_{10}(\text{Mfe}) = \frac{4559.5739}{\circ C + 273.16} - 6.7515$$

At the base material correlation time, the related material's relative thermal index is given by:

$$\log_{10}(77,228) = \frac{4559.5739}{(°C + 273.16)} - 6.7515$$

which can be calculated as 118.6°C. Using the procedures in 20.2.11, the material would be assigned a relative thermal index of 115°C.

20.3 Thermosetting molded materials

20.3.1 Thermosetting materials that are related to other materials evaluated under the aging program in the same manner and within the same limits as thermoplastic materials as described in 20.2.1 - 20.2.15 and Table 9.1 of UL 746A are also eligible for the abbreviated test program. In addition, because periodic variations are often necessary in the formulation of thermosetting materials in order to adapt to variable sources of supply and to adjust for variable molding conditions, it is acceptable if the limits specified in Table 9.1 of UL 746A are exceeded, provided that the same numerical compound designation is used and the conditions in 20.3.2 and 20.3.3 are met.

20.3.2 An abbreviated heat-aging test is to be conducted in accordance with the options provided in 20.2.5 or 20.2.15. Property, time, temperature, and percent retention of the property are to be selected based on information obtained in the long-time thermal-aging program.

20.3.3 Analytical measurements are to be used to ascertain that the materials have essentially the same formulation ingredients, proportions, and properties. Infra-red analysis and Thermogravimetry determinations are to be included. Differential Scanning Calorimetry may also be included where applicable.

Table 20.1
An example illustration for Two-Sample T-Test for Tensile Strength (MPa)

Α	В	С	D	E	F	G
No	Control (as received)	Control (2100 hours)	(Percent retention) _{Control}	Candidate (as-received)	Control (2100 hours)	(Percent retention) _{Candidate}
1	28.2	16.8	60.3	31.1	18.6	59.3
2	27.3	15.2	54.5	32.5	17.3	55.2
3	29.6	16.2	58.1	29.5	16.2	51.7
4	28.0	17.6	63.1	33.4	17.3	55.2
5	27.1	15.8	56.7	30.5	19.4	61.9
6	29.3	15.0	53.8	31.6	17.5	55.8
7	28.6	14.7	52.7	32.1	16.3	52.0
8	27.1	17.1	61.3	29.4	19.8	63.2
9	27.8	17.0	61.0	29.8	16.8	53.6
10	28.3	16.9	60.6	31.6	16.2	51.7
11	29.1	16.2	.58.1	30.6	17.4	55.5
12	29.0	15.8	56.7	31.9	16.8	53.6
13	26.5	14.9	53.4	33.4	18.6	59.3
14	27.8	14.0	50.2	32.4	19.1	60.9
15	28.2	16.7	59.9	31.7	18.2	58.1
16	28.0	16.3	58.5	30.7	19.0	60.6
17	27.8	17.7	63.5	31.6	18.0	57.4
18	26.4	16.2	58.1	33.7	17.2	54.9
19	28.1	15.4	55.2	32.5	18.3	58.4
20	29.1	14.8	53.1	32.7	19.2	61.3
21	28.4	14.9	53.4	30.1	20.4	65.1
22	26.8	16.2	58.1	29.4	19.3	61.6
23	27.5	17.3	62.1	31.5	19.1	60.9
24	28.3	15.1	54.2	30.6	20.4	65.1
25	26.7	15.7	56.3	31.7	18.2	58.1
26	27.5	16.8	60.3	32.8	18.8	60.0
27	27.5	15.2	54.5	30.1	17.8	56.8
28	26.8	14.8	53.1	29.6	20.1	64.1
29	29.0	17.2	61.7	31.1	16.8	53.6
30	26.5	16.8	60.3	30.7	19.0	60.6
Mean	27.88	_	57.43	31.34	_	58.18

NOTE: The percent retention values calculated in Columns D and G for 30 samples are based on the Mean Values of their respective as-received sample group.

p-Value of (Percent Retention)_{Control} and (Percent Retention)_{Candidate} calculated based on a two-sample, 2-tailed homescedastic (equal variance) T-Test is 0.443. Since this value is greater than 0.05 significance level, the degradation behavior of control and the candidate materials is not statistically different.

21 Aging, Specimen, and Check-Test Schedules

21.1 General

- 21.1.1 <u>Table 21.1</u> <u>Table 21.3</u> are for use in assisting manufacturers in formulating a long-time thermalaging program.
- 21.1.2 The schedules shown in <u>Table 21.1</u> <u>Table 21.3</u> are examples for demonstration purposes only. Specific aging temperatures, tests, specimen sizes, etc. are to be applicable to the specific polymer and end use. In most cases, five specimens per measurement are to be employed but, in some cases, ten specimens are needed.
- 21.1.3 The number of specimens tabulated is based on the presumption of attaining the property end point within the number of aging cycles indicated in the delayed-set schedules.
- 21.1.4 Described in 21.2.1 21.3.13, are particular test programs for materials of procedures of unusual nature that do not follow the general procedures shown in Table 21.1 Table 21.3.

21.2 Polypropylene

- 21.2.1 For polypropylene, it is observed that the occurrence of visible crazing indicates the severe and sudden loss of material properties. The thermal-aging procedure described in Table 21.1 Table 21.3 may be considerably reduced since surface crazing can be used as a preliminary indication of material-property loss. The quantity and sizes of samples required for a polypropylene thermal-aging program are described in Table 21.4.
- 21.2.2 Thermal aging is to be conducted at four oven temperatures as described in <u>Table 13.1</u>, for example 160, 150, 140 and 130°C (320, 302, 284, and 266°F). Samples are to be aged at all four temperatures for evaluation of the primary properties of tensile impact and tensile strength. Samples are to be aged at either of the two intermediate test temperatures for evaluation of the secondary properties of flammability and dielectric strength.
- 21.2.3 Ovens at each temperature are to be loaded with one set of test samples initially (set A). The second set of samples (set B) is to be placed in each oven at a later time than the initial batch (set A) in accordance with Table 21.5
- 21.2.4 Using the proof testing method described in 18.1, for each different sample configuration, thickness and test temperature, the property end point is to be determined by noting the time at which each initial set of test samples (set A) shows crazing on 10 percent of the total surface area of each specimen. When this crazing occurs, the oven time is to be recorded and all crazed samples are to be removed from the oven. When all the initial samples (set A) have crazed, the delayed samples (set B) and secondary-property samples are to be removed from the oven. Prior to property testing, of the delayed (set B) and secondary-property samples, the samples are to be conditioned in accordance with Table 21.1.
- 21.2.5 The proof testing method described in Exception No. 2 of <u>18.1</u> is to be used to determine the average log life for the 10 initial test samples (set A) for each different configuration and test temperature. The second set of tensile strength, tensile impact and dielectric strength samples (set B) shall retain at least 50 percent of the initial property value and the flammability classification shall not change.

21.3 Coating powders

21.3.1 The testing of coating powders to determine a relative thermal index for use as ground insulation in motors, transformers, bus bars, and the like, operating at higher than Class 105 temperatures, is covered in 21.3.2 - 21.3.13.

- 21.3.2 This subsection under coating powders is to be considered only as a guide for establishing a testing program, as specific details must be worked out for each material and end-use application. The tests are to include consideration of all variations in chemical composition, color percentage mix, molecular weight, etc.
- 21.3.3 The end-product evaluation is to result in the final judgment concerning the test performance (such as for insulation), constructional requirements (such as thickness), and other considerations, such as:
 - a) Normal and abnormal tests.
 - b) Additional abnormal tests necessitated by the specific polymeric material.
 - c) Effect of adjacent insulation on performance at points of material contact.
 - d) The general maximum voltage rating under this program is 600 volts. If higher voltages are a consideration, additional testing is necessary higher dielectric-strength potentials, resistance to partial discharge, etc.

Table 21.1
Conditioning before property measurement (Example)

Property	1111
Tensile or flexural strength,	Min 40 h exposure to 50 ±10 percent relative humidity at 23.0
Tensile, Izod, Charpy impact, or Dielectric strength ^a	±2.0°C (73.4 ±3.6°F)
Flammability (material rated V-2 or better)	Cooled in desiccators a minimum of 4 hours after oven exposure
^a The surrounding medium for the dielectric strength test shoul D149	d be air, or oil using shrouded electrodes in accordance with ASTM

Table 21.2 Typical aging schedule (Example)

Material	Thickness mm	77.	Aging temp	erature, °C			Cycle peri	ods, days ^a	
	ILA	Α	В	С	D	Α	В	С	D
Candidate	3.2	130	140	150	160	28	14	7	3
(proposed)	1.6	_	140	150	_	_	14	7	_
(proposed)	0.8	_	140	150	-	_	14	7	-
Control (known)	3.2	130	140	150	160	28	14	7	3

Table 21.3 Number of specimens required for thermal aging (example)

	Те	st			Speci	imens	
Test material	Property	Method	Thickness mm	Number per set ^c (B - L)	Number for initial tests ^c	Minimum number for all temperatures	Total ^{b,d}
Candidate (proposed)	Tensile or flexural strength	UL 746A	3.0 1.5 0.75	5 5 5	10 10 10	220 110 110	230 120 120
	Tensile, Izod, or Charpy impact	UL 746A	4.0 ^e	5	10	220	230
	Dielectric strength	UL 746A	0.75	5	10	220	230
	Flammability (materials rated V-2, VTM-2, or better)	UL 94	MTª	10	10	160	170
Control (known)	Tensile or flexural strength	UL 746A	3.0	5	× 60	220	230
	Tensile, Izod, or Charpy impact	UL 746A	4.0 ^e	5 IIP	10	220	230
	Dielectric strength	UL 746A	0.75	11/05	10	220	230

^a MT represents the minimum thickness evaluated.

^b It is recommended to prepare samples in excess of this total in case there is a dispute of the results and a reevaluation is considered necessary.

^c See Table 16.1.

^d For example, 5 specimens per 5 initial sets (B T) plus 5 specimens per 3 delayed sets (G – I) plus 5 specimens for 3 extra sets (J – L) equals 55 specimens, multiplied by 4 temperatures equals 220 specimens plus 10 unaged (set A) specimens equals 230 total specimens.

^e Impact test specimens in a thickness less than 4.0 mm but greater than or equal to 3.0 mm may also be used but may increase the risks of dimensional deviations or handling problems after thermal aging.

Table 21.4

Number of specimens required for a typical polypropylene thermal aging program

	Tes	st	Thickness mm			Spe	cimens	
Test material	Property	Method	ASTM	ISO	Number per set	Number for initial tests	Number for all temperatures (sets A and B)	Total
Candidate (proposed)	Tensile strength	UL 746A	3.2	4.0	10	10	80	90
	Tensile or Charpy impact	UL 746A	3.2 1.6	4.0 2.0	10 10	10 10	80 40	90 50
	Dielectric strength	UL 746A	1.6	2.0	5	10	20	30
	Flammability (materials rated V-2 or better)	UL 94	MT ^a		5	10	6B 120 L	30
Control (known)	Tensile strength	UL 746A	3.2	4.0	10	10	80	90
	Tensile or Charpy impact	UL 746A	3.2	4.0	10	O 10	80	90
^a MT represe	nts the minimum	thickness eval	uated.		., 0			

Table 21.5

Delay time for insertion of verification samples in polypropylene aging programs

Aging temperature °C (°F)	Delay time to insert second sample set (Set B) in oven after start of program, days
160 (320)	3
150 (302)	7
140 (284)	14
130 (266)	28

21.3.4 The test specimens to be used for motor or transformer ground insulation are to be steel U-channels of the shape and size shown in Figure 21.1. The scale on the interior surface of the specimen is to be removed by means of sandblasting or an acid rinse followed by a water rinse, an alkaline rinse, and a final water rinse. Each specimen is to be machined as indicated in Figure 21.1 to a 32-microinch (810-micrometer) finish. The specimens are then to be coated with powder in the thickness specified by the manufacturer using a typical process. The powder is then to be cured as advised by the manufacturer. One end of the specimen is to be left uncoated for attaching the specimen to a vibration machine and also for making an electrical connection during the dielectric-strength tests. Prior to aging, all specimens are to be subjected to a screening test in order to remove defective units.

Exception: Ground tool-steel bits as illustrated in Figure 21.2 may be employed in place of U-channels.

Table 21.6
Typical Number of specimens required for thermal aging of film materials

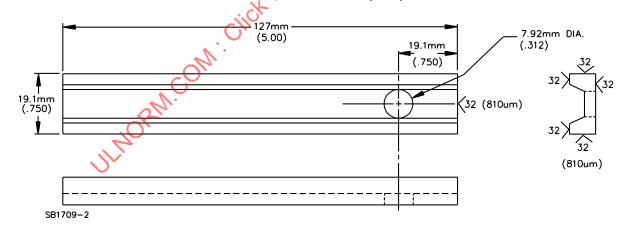
Test			Specimens				
Test material	Property	Method	Thickness mm	Number per set	Number for initial tests	Number for all temperatures	Total ^{b,c}
Candidate (proposed)	Tensile strength	UL 746A	0.127 MT ^a	5 5	10 10	220 110	230 120
	Dielectric strength	UL 746A	MT ^a	5	10	220	230
	Flammability (materials rated VTM-2 or V-2 or better)	UL 94	MT ^a	10	10	160	170
Control (known)	Tensile strength	UL 746A	0.127	5	10	220	230
	Dielectric strength	UL 746A	MT ^a	5	10	220	230

^a MT represents the minimum thickness evaluated.

Figure 21.1

Specimen dimensions

All dimensions in mm (inch)



Material: American Iron and Steel Institute Type C1020 steel, U-channel, standard 19.05 by 7.94 by 3.18 mm (3/4 by 5/16 by 1/8 inch) bar stock. Modified as follows:

- 1) The outer surfaces and the end of the specimen that is to be coated are to be machined to a 810-micrometer (32-microinch) finish.
- 2) The finished dimensions are not critical.
- 3) Burrs are to be removed without rounding the edges.

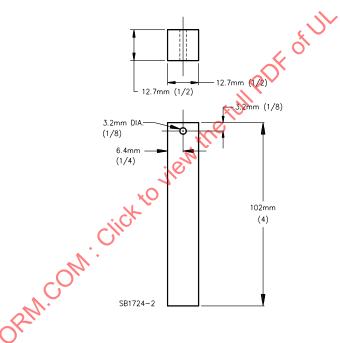
b It is recommended to prepare samples in excess of this total in case there is a dispute of the results and a reevaluation is considered necessary.

^c For example, 5 specimens per 5 initial sets (B − F) plus 5 specimens per 3 delayed sets (G − I) plus 5 specimens for 3 extra sets (J − L) equals 55 specimens, multiplied by 4 temperatures equals 220 specimens plus 10 unaged (set A) specimens equals 230 total specimens.

- 21.3.5 The test specimens to be used for evaluating integral bus-bar insulation systems are to be copper and/or aluminum specimens of the size and shape shown in Figure 21.2.
- 21.3.6 To determine an initial dielectric-strength level, specimens are to be selected at random and subjected to a voltage breakdown test. To prevent flashover, the unaged specimens may need to be provided in a length greater than 127 mm (5 inch). One electrode of the tester is to be connected to the uncoated end of the specimen and approximately 50.8 mm (2 inch) of the coated end of the specimen are to be immersed into a 10-percent-salt-water solution, which is the other electrode of the tester. The voltage from the tester is to be increased at a rate of approximately 500 volts per second until breakdown occurs. The average breakdown value of the ten specimens is to be recorded as the initial breakdown voltage of the material. At least ten specimens are to be placed in each of four ovens, the temperatures of which are to be determined by the manufacturer. The highest temperature is to result in a life of at least 500 hours while the lowest temperature is to result in a life of at least 5,000 hours.

Figure 21.2

Alternate tool-steel and bus-bar test specimens All dimensions in mm (inch)



NOTE -

Substrate material is to be ground square to within 0.01 mm (0.001 inch) with sharp (no measurable radius) edges. 810-micrometer (32-microinch) finish or four long sides.

- 21.3.7 To provide approximately equal exposures to the other conditionings and to more accurately determine the property end point , the heat aging time per cycle is to be less for the higher aging temperatures for example:
 - a) Highest oven temperature 1 or 2 days per cycle
 - b) Next oven temperature 2 to 4 days per cycle
 - c) Next oven temperature 4 to 14 days per cycle
 - d) Lowest oven temperature 3 to 7 weeks per cycle

- 21.3.8 At the end of each exposure in the oven, all samples are to cool to room temperature for approximately 1 hour. All samples are then to be subjected to a cold shock for 1 hour. If the coating resin is intended for outdoor applications, the temperature of the cold box is to be minus 20.0 $\pm 2.0^{\circ}$ C (minus 4.0 $\pm 3.6^{\circ}$ F). If the coating resin is intended for indoor applications only, the temperature of the cold box is to be $0.0 \pm 2.0^{\circ}$ C ($32.0 \pm 3.6^{\circ}$ F).
- 21.3.9 Following the cold shock, all samples are to stand for 1 hour at room temperature. All samples are then to be subjected to a vibration test consisting of 10,000 cycles of vibration at an acceleration of 7 G's. If the motion of the specimen during the vibration test is simple harmonic, the maximum peak-to-peak deflection is to be 0.97 mm (0.038 inch), if the frequency of vibration is 60 hertz. The direction of the vibration is to be parallel to the shortest dimension of the specimen and orthogonal to the largest flat surface of the specimen.
- 21.3.10 Following the vibration test, all samples are to be subjected to a humidity test for a period of 24 hours at $25 30^{\circ}$ C ($77 86^{\circ}$ F) with the relative humidity adjusted to 95 100 percent.
- 21.3.11 Within 1 hour after the humidity test, all specimens are to be subjected to a proof voltage test. The applied voltage is to be raised from zero at a rate of approximately 500 volts per second until the voltage reaches a value equal to 50 percent of the initial breakdown voltage of the material. This value of the voltage is to be maintained for 5 seconds, and the voltage is then to be removed from the specimen. Specimens that do not break down during this test are to be returned to the oven for further testing.
- 21.3.12 The aging-test program is to be continued until all specimens have exhibited breakdown. The time to breakdown for each specimen is to be recorded.
- 21.3.13 The data is to be evaluated by the proof-testing method described in Proof Testing, Section 18.

22 Fixed Time Sampling Method

22.1 General

- 22.1.1 As an alternate to the Fixed Temperature Method in Sections $\underline{13} \underline{21}$, the sampling method described in this section may be used to conduct the long-term heat aging program. The primary difference between the methods is in the sampling technique employed. Since both methods rely upon a data analysis of the degradation of samples at various temperatures and using specific time intervals, the results of the tests would be expected to be similar regardless of which of the two methods is selected.
- 22.1.2 Section 14, Selection of Control Material, and Section 15, Specimens, are applicable to the Fixed Time sampling method.
- 22.1.3 This sampling method was developed with the objective of completing most evaluations within approximately 5,000 hours of testing. This is accomplished through a more intensive selection of aging temperatures in the early portion of the program (the screening procedure) and by using the aging temperatures as the dependent variables while aging interval are the controlled variable.
- 22.1.4 To determine the performance characteristics of the material, a series of specimens is to be tested for property retention levels following fixed time frame aging intervals. The aging temperatures for the Fixed Time Frame Method are normally not established until after the screening procedure described in Section 22.2 is completed.

22.2 Screening procedure

22.2.1 As a general guideline in selecting the temperatures to be used for conditioning of the thermal aging specimens, select four or more aging temperatures starting approximately 40°C above the expected