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Designation: C 1303 – 9500

Standard Test Method for Estimating the Long-Term Change in the Thermal Resistance of Unfaced Rigid Closed-Cell Plastic Foams by Slicing and Scaling Under Controlled Laboratory Conditions^{1,2}

This standard is issued under the fixed designation C 1303; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for estimating the long-term change in thermal resistance of unfaced rigid closed-cell

² ISO/TC163/SC2/WG7 is also developing a standard to address the aging of unfaced rigid closed_cell plastic foams. This document is entitled," Determination of the Long-Term Thermal Resistance of Closed_Cell Cellular Plastic Thermal Insulation."

¹ This test method is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurement.

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plastic foams by reducing the material thickness to accelerate aging under controlled laboratory conditions (1-3).³

1.2 This test method is limited to unfaced, homogeneous materials (see 3.2.4). It may be applied to a wide range of rigid closed cell plastic foam types, including but not limited to, polystyrenes, polyurethanes, polyisocyanurates, and phenolics produced in board form, foamed-in-place, or spray-applied applications. No specific procedures are detailed in this test method to address the effects of permeable or impermeable facings or skins, manufactured thickness, orientation, manufacturing process, density, quality, the influence of structures or containments, or the end-use environmental conditions on internal cell gas composition. The user of this test method shall consider if these parameters limit the use of this test method for a specific application.

1.3 This test method utilizes standard test procedures for measuring the thermal resistance of insulation materials. resistance. Periodic measurements are performed on specimens to observe the effects of aging. Specimens of reduced thickness are used to shorten the time required for these observations. The results of these measurements are coupled with a scaling factor to estimate the thermal resistance of the material under evaluation for other thicknesses as a function of time.

1.4 This test method specifies methods of specimen preparation, procedures for determining the specimen effective diffusion thickness (see 3.2.3), and precautions for determining the thermal resistance of thin specimens.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 This test method should be used to measure and describe the relative change in thermal resistance of rigid closed_cell plastic foams under controlled laboratory conditions. It should not be used to describe or appraise the performance of these materials or products under actual use conditions. With continuing development, results from this test method may be used as an element in an assessment which takes into account all of the factors that are pertinent to an estimation of the thermal performance of these materials. Critical elements of this assessment are presently not available. See 1.2.

1.7 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- C 168 Terminology Relating to Thermal Insulating Materials⁴
- C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus⁴
- C 236 Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box⁴
- C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus⁴
- C 578 Specification for Rigid, Cellular Polystyrene Thermal Insulation⁴
- C 591 Specification for Unfaced Preformed Rigid Cellular Polyisocyanurate Thermal Insulation⁴
- C 976 Test Method for Thermal Performance of Building Assemblies by Means of a Calibrated Hot Box⁴
- C-984 Specification for Perlite Board and Rigid Cellular Polyisocyanurate Composite Roof Insulation⁴
- C 1013 Specification for Faced Rigid Cellular Polyisocyanurate Roof Insulation⁴
- C 1029 Specification 1029 Specification for Spray-Applied Rigid Cellular Polyurethane Thermal Insulation⁴
- C 1045 Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements⁴
- C-1050 Specification for Rigid Cellular Polystyrene-Cellulosic Fiber Composite Roof Insulation⁴
- C 1114 Test 1114 Test Method for Steady-State Thermal Transmission Properties by Means of the Thin-Heater Apparatus⁴
- C 1126 Specification for Faced or Unfaced Rigid Cellular Phenolic Thermal Insulation⁴
- C 1289 Specification for Faced Rigid Cellular Polyisocyanurate Thermal Insulation Board⁴
- D 2856 Test Method for Open-Cell Content of Rigid Cellular Plastics by the Air Pycnometer⁵
- E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process⁶

2.2 Other Standard:

³ The boldface numbers in parentheses refer to the list of references at the end of this standard.

⁴ Annual Book of ASTM Standards, Vol 04.06.

⁵ Annual Book of ASTM Standards, Vol 08.02.

⁶ Annual Book of ASTM Standards, Vol 14.02.

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HH-I-1972 Federal Specification, Insulation Board, Faced, Polyurethane or Polyisoeyanurate⁷

3. Terminology

3.1 Definitions—For definitions of terms and symbols used in this test method, refer to Terminology C 168.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *aging*, <u>v</u>—the change in thermophysical properties of rigid closed_ cell plastic foam with time primarily due to changes in the composition of the gas contained within the closed cells.

3.2.2 effective diffusion coefficient, <u>n</u>—a material property that relates the rate of gas transport to the gas partial pressure gradients across the material of a given thickness at a given temperature. The term—""effective" is used to differentiate between diffusion coefficients of individual gases and gas mixtures. describe mass transport by several mechanisms.

3.2.3 *effective diffusion thickness, n*—the geometric thickness minus two times the thickness of damaged surface layer (TDSL). See *thickness of damaged surface layer (TDSL)*.

3.2.3.1 *Discussion*—If aging is limited to a single surface due to encapsulation or surface sealing, the effective thickness would be the geometric thickness minus the TDSL.

3.2.4 homogeneous material, <u>n</u>—a material with a variation of less than 10% in the slope of the primary stage thermal resistivity versus time $\underline{\tau}^*$ results, for specimens within a specific sample. See 7.1.1 and 7.1.2. As more data becomes available, this description will be refined.

3.2.5 <u>pnormalized thermal</u> <u>ryesistagnce</u>—that portion of the aging process where changes in thermophysical properties are primarily influenced__thermal resistance divided by the diffusion of air components into the rigid closed cell plastic foam. intial thermal resistance.

3.2.6 *scaling factorprimary stage*—the rat portion of the square of the material thickness and the square of the test specimen thickness (see 5.2.4). This ratio represents the acceleration rate that is being applied to the aging process where changes in thermophysical properties are primarily influenced by the diffusion of air components into the rigid closed_cell plastic foam because of thickness differences. See Ref (1) for a detailed derivation of scaling factor. foam.

3.2.7 *secondary stagescaled time*—that portion of the aging process where changes in thermophysical properties are primarily influenced—time divided by the diffusion square of blowing agent(s) from the rigid closed cell plastic foam. specimen thickness.

3.2.8 <u>service lifescaling factor, n</u>—the anticipated period square of time that the material thickness divided by the square of the test specimen thickness (see 5.2.4). This ratio represents the acceleration rate that is expected being applied to maintain claimed thermophysical properties. The service life may be dependent on the s aging perociess of a rigid closend-cell plastic foam because of thickness differences. See Ref (1) for app detailed derivation of scaling factor.

3.2.9 *thicknesssecondary stage*, *n*—that portion of damaged surface layer (TDSL)— the average thickness of surface cells, on one surface, that aging process where changes in thermophysical properties are either destroyed (ruptured or opened) during primarily influenced by the preparation diffusion of test specimens or were originally open due to blowing agent(s) from the manufacturing process. rigid closed_cell plastic foam.

 $3.2.10 \ time-averaged thermal resistances ervice life, n$ —the thermal resistance anticipated period of a material of given thickness averaged over a specified time that the material is expected to maintain claimed thermophysical properties. The service life may be dependent on the specific end-use application.

3.2.11 *thickness of damaged surface layer (TDSL)*, *n*—the average thickness of surface cells, on one surface, that are either destroyed (ruptured or opened) during the preparation of test specimens or were originally open due to the manufacturing process.

3.2.12 *time-averaged thermal resistance*, *n*—the thermal resistance of a material of given thickness averaged over a specified time period.

<u>3.2.13</u> transition point (T_v) , <u>n</u>—the estimated age of a rigid closed_cell plastic foam when the aging process switches from the primary to secondary stage (see Figs. 1 and 2).).



3.3 Symbols: Symbols:



<u>R</u> TDSL τ^* qqgqrqs

= surface area of Test Method D 2856 specimen, m^2 Α D \equiv diffusion coefficient, m²/s partial pressure, atm = Ð thermal resistance, $(m^2 \cdot K)/W$ R = R^* normalized thermal resistance= R_t/R_0 Ξ $\overline{R}_{\underline{0}}$ initial thermal resistance Ξ \underline{R}_a =estimated time-averaged thermal resistance, (m²·K)/W thermal resistance on t^{th} day, $(\text{m}^2 \cdot \text{K})/\text{W}$ R = time, days = t service life, days = t_m transition point, days average thickness of damaged surface layer, m $t_{t}TDSL =$ $TDSL_{\tau}^* =$ average thickness of damaged surface layer, m scaled time, sec/mm² bulk geometric specimen volume of Test Method D 2856 specimen, m³ V_b = = closed_cell volume of the specimen of Test Method D 2856 specimen, m^3 V_c ΔX_{geo} = geometric thickness of thermal resistance specimen, m ΔX_{eff} = effective diffusion thickness of thermal resistance specimen, m ΔX_{1} ΔX sample (material) thickness, m = ΔX_2 = specimen thickness, m <u> ⊁specime</u>n thickness,m = apparent thermal conductivity, W/(m·K) q $\lambda_{\underline{g}\underline{heat}}$ flux, W/m^2 rrent thermal conductivityheat flux due to the cell gas-mixture, W/(m-K) q_{e} $\frac{\lambda_o}{\lambda_o}$ mixture,W/m² $\underline{q_r}$ ntheat flux due to thermal-conductivity , radiation, W/m2 V/(m-K)-the solid polymer,W/m2 <u>qs</u> ityheat flux due to ther λ5 standard deviation

4. Significance and Use

4.1 Rigid closed_cell plastic foam insulations are produced by foaming various polymers. As manufactured, the cells of the foam usually contain their highest percentage of blowing agent and the lowest percentage of air components. As time passes, the relative concentrations of these gases change due primarily to diffusion, resulting in a reduction of the thermal resistance of the foam due to an increase in the thermal conductivity of the resultant cell gas mixture.

Note 1—The discussions in Sections 4 and 54 and 5 assume that the blowing agent(s) is a gas whose apparent thermal conductivity and effective diffusion coefficient are both lower than those of the air components. If the blowing agent diffuses faster than the air components, definitions of the stages of aging would require modification and any discussions regarding diffusion rates would need to be changed. However, the test procedures are applicable in either case.

4.2 The change in thermal resistance due to the phenomena described in 4.1 usually occurs over an extended period of time at room temperature. Information regarding changes in the thermal resistance of these materials as a function of time is required so that decisions regarding formulations, production, and comparisons with other materials can be made. Ideally, aging curves and estimated time-averaged thermal resistance data for the expected service life should be available after as short a period as possible.

4.3 Specifications C 578, C 591, C 984, C 1013, C 1029, C 1050, <u>C 1126</u> and C 1126, as well as Fed. Spec. <u>HH-I-1972</u> <u>C 1289</u>, on rigid closed-cell plastic foams, indicate that this decrease in thermal resistance occurs over an extended period of time at room temperature. However, these standards currently require that freshly manufactured foams be measured for thermal resistance after conditioning at $23 \pm 1^{\circ}$ C ($73 \pm 2^{\circ}$ F) for 180 ± 5 days from the time of manufacture, or at $60 \pm 1^{\circ}$ C ($140 \pm 2^{\circ}$ F) for 90 days. These standards do not currently specify long-term or time-averaged thermal resistance criteria.

4.4 The procedure described in this test method requires that the material characteristics of the thin specimens approximate those of the material under investigation. In particular, the specimens of reduced thickness must have the same effective diffusion coefficient and initial cell gas content as those of the full thickness material, and that one-dimensional diffusion dominates, limiting the application of this test method to unfaced homogeneous materials as defined in 3.2.4.

4.4.1 When the thin specimen does not effectively represent the average behavior of the material, the results obtained by this test method may have a limited value.

4.5 This test method addresses three separate elements relating to the aging of rigid closed–cell plastic foams.

4.5.1 *Specimen Preparation*—Techniques for the preparation of thin flat specimens and the measurement of specimen thickness are discussed, along with their limitations.

4.5.2 *Measurement of the Thermal Resistance*—In principle, any of the referenced test methods for the determination of thermal resistance are suitable. These include Test Methods C 177, C 236, C 518, C 976, and C 1114, used in conjunction with Practice C 1045. Of these test methods, the heat flow meter apparatus, Test Method C 518, is preferred.

4.5.3 Interpretation of Data—Procedures are detailed for utilizing periodic short-term thermal resistance data to estimate long-term changes in the thermal resistance of the material. Examples are provided in Annex A1.

4.6 The procedure outlined in this test method can be used to produce a characteristic aging curve (relationship between the thermophysical properties with time). This relationship has been used by researchers to calculate effective diffusion coefficients (2, 3).

5. Background and Theory

5.1 The Aging Process:

5.1.1 During the service life of a rigid closed_cell plastic foam, air components diffuse into the cells, and the blowing agent diffuses out of the cells or partially dissolves into the polymer matrix. Each process occurs at a rate that depends on the type of polymer, the foam structure, the temperature, the gas type, and its pressure (1).

5.1.2 In general, as the inward diffusion of air components is much faster than the outward diffusion of the captive blowing agent, the aging process comprises two stages. During the primary stage, the cell gas composition changes at a significant rate because of the rapid diffusion of air components into the cell and the outward diffusion of all diffuse blowing agents, if present; so too does the thermal resistance of the material.

5.1.2.1 If carbon dioxide or other rapidly diffusing gases are used as blowing agents or are generated during foam manufacture, their outward diffusion rate will usually exceed the entry rate of air components during the primary stage.

5.1.3 Once the diffusion of air components nears completion, the thermal resistance of the material changes more slowly. The thermal resistance continues to change, however, due to continuing diffusion of the blowing agent from the cells. This stage is defined as the secondary stage.

5.1.4 Upon completion of the secondary stage of aging, the thermal resistance of the material no longer changes with time.

5.1.5 A number of researchers studying aging have depicted their thermal performance data (thermal conductivity, thermal resistance, thermal resistivity) for rigid closed_cell plastic foams as a function of time using the following functional forms (4-11).

 $-R = F\{\log t\} \text{ or } \log R = F\{t\}$	(1)
$R^* = F \{ \log (D \times t/(\Delta X)^2) \} \text{ or } $	(1)

Figs. 1 and

$\log (R^*) = F \{ D \times t / (\Delta X)^2 \}$

This formulation is the basis for the scaled time variable, τ^* . This variable does not include the diffusion coefficient, *D*, but because the diffusion coefficient for each gas is a constant at constant temperature, the functional form of the relationship is <u>unaffected</u>. Figs. 2 and 3, and Fig. 1 are examples of aging curves for a rigid closed_cell plastic foam (**127**). The inflection between the two stages of the aging process is defined as the transition point. A second transition may occur, depending on the combination

if all of the blowing agent is u replaced within the cells by air. After all the cells are filled with air, the thermal performance should remain constant.

5.2 Use of Thin Specimens:

5.2.1 The apparent thermal conductivity of heat flux, q, passing through a rigid closed-cell plastic-foam, λ , foam can be approximately expressed as the sum of the apparent thermal conductivities heat flux due to radiation $(\lambda_r), (q_r)$, due to the gas mixture $(\lambda_g), (q_g)$, and due to the solid polymer $(\lambda_s, (q_s), (132))$.

$$\lambda = \lambda_r + \lambda_g + \lambda_s \tag{2}$$

$$q = q_r + q_g + q_s \tag{2}$$

5.2.2 It is assumed that the <u>sum of the</u> values of $\lambda_{r q_{i}}$ and $\lambda_{s and q_{s}}$ dodoes not change significantly with time-as even though the gas content within the cells changes. Then, Eq 2 implies that the aging process can be studied by exclusively investigating the change in the gas thermal conductivity, heat flux due to the gas, and $\lambda_{g q_{s}}$ can be determined by studying the change in <u>molecular</u> concentration (partial pressure) of the cell gas components as a function of time. The governing parameters controlling the changes in the partial pressure, p, pressures of the gas components are their effective diffusion coefficients D, the thickness ΔX and time t (1). In order to accelerate the aging process, either the diffusion coefficients can be increased or the thickness reduced.

5.2.3 Diffusion coefficients can be increased by raising the temperature, but this method is not recommended for the following reasons (13). The amount of acceleration achievable by this test method is limited because the diffusion coefficients are typically not a strong function of temperature. A second limitation is that a specific increase in temperature does not equally change the diffusion coefficients of all the gases involved in the aging process. Another possible limitation is that elevating the temperature could damage the cellular structure of the foam (14). The amount of acceleration achievable by this test method is limited because the diffusion coefficients are typically not a strong function of temperature. A second limitation is that a specific increase in temperature does not equally change the diffusion coefficients of all the gases involved in the aging process. A second limitation is that a specific increase in temperature does not equally change the diffusion coefficients of all the gases involved in the aging process. A third possible limitation is that elevating the temperature could damage the cellular structure of the foam (15).

5.2.4 Reducing specimen thickness can increase the aging rates and does not expose the material to potentially damaging or unrealistic conditioning at elevated temperatures. For a material satisfying the requirements of constant *D* and initial *p*, the same value of the ratio $t/\Delta X^2$ will yield the same value of *p*, and therefore the same values of $\lambda \underline{q}_g$ and $\lambda \underline{q}$. Therefore, the thermal resistance of a specimen of thickness ΔX_1 at time t_1 can be determined after conditioning a specimen of effective thickness ΔX_2 over a time interval t_2 . The time interval t_2 can be calculated by:

$$t_2 = t_1 \times (\Delta X_2 / \Delta X_1)^2 \tag{3}$$

The ratio $(\Delta X_2/\Delta X_1)^2$ is defined as the scaling factor. See Annex A1 for an example calculation and Ref (1) for the complete derivation.

5.3 Utilization of Thermal Resistance Data:

5.3.1 *Calculation of Time-Averaged Thermal Resistance*—Assuming that R_t is the thermal resistance of a rigid closed_cell plastic foam on the t^{th} day, R_a is the time-averaged thermal resistance, and t_m is the intended service life of the rigid closed_cell plastic foam in days, then:

$$\underline{R_a} = (1/t_m) \int_{-\tau}^{t_m} R_t \, dt \tag{4}$$

$$R_a = t_m / \{ \int_{0}^{t_m} (1/R_t) dt \}$$
(4)

5.3.1.1 Periodic measurements of the thermal resistance of the thin specimen will supply the necessary data to solve Eq 4. Care must be taken to guarantee that there is sufficient data to characterize accurately both the primary and secondary stages. See A1.2 for an example of the necessary steps to perform this analysis.

NOTE 2—The frequency of taking measurements is impacted by the type of material being analyzed and the thickness of the test specimen. Materials that age rapidly and extremely thin test specimens require many measurements over a short period of time to guarantee that sufficient data are generated to define the primary stage of aging. When evaluating a new material or a material whose aging characteristics are unknown, it is recommended that a trial series of experiments be undertaken to determine the testing frequency.

5.3.1.2 When determining the time-averaged thermal resistance, R_a , R_t shall have been determined over a period of scaled time that includes t_m , the service life of the full thickness specimen. No extrapolation shall be allowed.

6. Apparatus

6.1 Thermal resistance test apparatus used for this test method shall meet the following requirements:

6.1.1 *Conformance to Standards*—The apparatus shall conform to all of the requirements of the selected test method. See 4.5.2. 6.1.2 *Size and Error*—The apparatus shall be capable of testing specimens with a thickness of 5 mm (0.2 in.) with an estimated error in thickness of not more than 2 % attributed to errors in plate flatness and parallelism. Larger apparatus command tighter tolerances in flatness and plate parallelism to satisfy this requirement. When using test methods that require calibration, additional uncertainties due to mismatch in the thermal resistances of the specimen and calibration sample shall also be considered.

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NOTE 3—Due to the limited thickness of the test specimen, its thermal resistance may be significantly less than that of the calibration specimen. Experiments that verify that the calibration is insensitive to heat flux level should be undertaken prior to testing the thin specimens.

6.1.3 *Temperature*—The apparatus shall be capable of tests at a mean temperature of $24 \pm 2^{\circ}C$ ($75 \pm 4^{\circ}F$) with a temperature difference of 10 to $28^{\circ}C$ (18 to $50^{\circ}F$).

Note 4—The use of temperature differences within the range of 10 to 28° C (18 to 50° F) may result in a variation in the measured properties. A temperature difference of 22° C (40° F) is recommended.

Note 5—Some users of this test method recommend that the temperature difference across the specimen be a function of the thermal resistance of that specimen. A target temperature, ΔT , can be calculated from $\Delta T = (a)(R)$, where *a* is equal to $32^{\circ}C/(m^2 \text{ K/W})$ (10°F/(h ft² F/Btu)).

6.1.4 *Humidity*—The humidity within the test apparatus shall be maintained low enough to prevent condensation from forming within the specimen or on the cold plate(s) of the apparatus.

6.2 Specimen preparation equipment that has successfully been used to prepare thin specimens is listed in 6.2.1-6.2.43. Reference (5) summarizes these techniques and compares their effectiveness. This listing is not intended to preclude the development or use of any other demonstrated methods of specimen preparation.

6.2.1 High Speed Band-saw, with a fine-tooth 1 tooth/mm (24 teeth/in.) blade.

6.2.2 Surface Grinder or Planer.

6.2.3 Combination Lathe/Motor-driven Meat Slicer.

6.2.4 Hot Wire Cutter.

6.2.5 Surface Damage—Equipment for preparing thin specimens shall be selected based on the equipment's ability to reproduce the amount of surface damage (open cells) created in the preparation process. Small variations in surface damage can yield significant variability in the data obtained from the thin specimen.

Note 6—Different specimen preparation equipment may be required to prepare thin specimens from generically different rigid closed_cell plastic foams.

6.2.65 *Thickness Uniformity*—The equipment used to prepare specimens shall be capable of producing specimens that are uniform in thickness. For specimens less than 300 mm (12 in.) square in cross section, a thickness survey consisting of at least nine measurements shall confirm that the specimen thickness does not vary by more than 2 % from the average thickness. Larger specimens shall require additional measurements; a minimum of 50 measurements/m² (5 measurements/ft²) shall be performed.

<u>6.2.6 *Hot-Wire*</u>Use of a hot-wire cutter is discouraged because it can produce a surface skin. For further discussion, please see 11.3 and Note 16.

6.3 The apparatus used to measure the effective diffusion thickness of the specimen shall be as specified in Test Method D 2856 or shall have demonstrated equivalent performance. See Ref (165) for a description of an acceptable alternative apparatus.

NOTE 7—The accuracy of Test Method D 2856 may be insufficient when used to determine the effective diffusion thickness of some thin specimens. The uncertainty associated with this procedure shall be considered when selecting the geometric thickness of the thin test specimen. The requirements of 8.1.3 shall be used as a guideline to ascertain whether Test Method D 2856 is adequate for the selected specimen thickness.

NOTE 8— When using Test Method D 2856, it is recommended that an average of the half-atmosphere and two-atmosphere methods be used. The two-atmosphere method is likely to cause cell damage in low density materials; for these materials, the half-atmosphere method should be used exclusively.

NOTE 9— At a minimum, equivalency between Test Method D 2856 and the proposed alternative shall be demonstrated by direct comparison of the two procedures.

7. Sampling

7.1 Since the data analyses utilized in this test method assume that the reduced-thickness specimen maintains the same diffusion coefficients and initial cell gas content as the material, care shall be taken during the sampling process to guarantee these conditions are met. Failure to satisfy this requirement prevents the generalization of data generated by this test method to the material.

7.1.1 For the purpose of this test method, multiple specimens removed from the core and surfaces will be considered homogeneous if the slopes of the thermal resistivity versus time τ^* plots do not vary by more than 10 %. In some cases, materials may have sufficient uniformity such that sampling from the core may be considered representative.

7.1.2 The application of this test method to materials that are not homogeneous has limited value; the derived properties shall not be applied to the material or for specifications purposes.

7.2 Specimens shall be prepared as soon as possible after production so that the cell gas content within the sample is representative of initial conditions. Refer to the appropriate material specification for additional guidance regarding the appropriate sampling time.

7.2.1 An alternate method is to prepare specimens from the center of a full thickness section; the center may still have a cell gas content that is representative of the section at the time of manufacture. Reference (1) discusses this issue in detail.

8. Specimen Preparation

8.1 Thermal Resistance Specimens:

8.1.1 From full-thickness material, remove a full thickness section whose cross-sectional area is large enough to fully cover the plate area of the test apparatus. It is recommended that a single specimen be prepared from each section.

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Note 10-Additional material is required for closed cell volume measurement specimens. See 8.2.1.

8.1.2 For the purpose of this test method, the minimum thickness shall be limited such that the TDSL (see 9.1) is no more than 5 % of the geometric thickness. To minimize the effects of specimen preparation on thermal performance, it is recommended that the specimen be at least 20 average cell diameters thick. Specimens 10 mm (0.39 in.) thick have been used extensively. However, different material types may require different thicknesses because of the amount of surface damage that will be obtained during the preparation process.

8.1.3 Reduce the thickness of this section by grinding, planing, or cutting thin layers from each major surface. It is recommended that multiple cuts or passes be performed on alternate surfaces to reduce the possibility of the specimen warping. Patience is strongly recommended for these operations.

NOTE 11—Different specimen preparation techniques have been used that satisfy the requirements of 6.2.5<u>4</u> and 8.1.3. Specifically, planing has been successfully employed to prepare specimens of polyisocyanurate foam and hot wire cutters have been used in the preparation of polystyrene foam specimens. foam.

8.1.4 The minimum number of replicate specimens to be tested shall be selected so that there is confidence that the average results from these sliced specimens are representative of the material undergoing testing; at least five sets of thermal resistance specimens per material-is are recommended. For additional guidance, refer to Practice E 122.

8.2 Closed Cell Volume Measurement Specimens:

8.2.1 Utilizing the procedure detailed in 8.1, prepare test specimens with dimensions that are required for the closed cell volume measurement apparatus. These specimens should be prepared from sample material taken adjacent to the thermal resistance specimen sections (8.1).

8.2.2 Since most closed cell volume measuring apparatus utilize small specimens, it is likely that the volume measurement will be performed on specimens that will not be measured for thermal resistance. It is therefore recommended that the user of this test method verify that the specimen preparation method produces specimens with a reproducible amount of surface damage by preparing and measuring at least five specimens prior to the general utilization of the preparation technique.

9. Test Procedures

9.1 Measurement of Specimen Effective Diffusion Thickness:

9.1.1 *Scope*—This test method covers the determination of the effective diffusion thickness of the thermal resistance specimen. 9.1.2 *Significance and Use*—The specimen preparation technique employed by this test method destroys the closed cell integrity of the surface cells. Because the specimens used to perform thermal resistance measurements are relatively thin, these damaged surface cells may account for an appreciable percentage of the total specimen thickness.

9.1.3 Procedure:

9.1.3.1 Measure the cross section and thickness and weigh the closed cell volume-measurement specimen to within the tolerances required by the measurement method (see 6.3). Compute the specimen geometric volume, V_b , and total surface area, A.

9.1.3.2 Measure the specimen closed cell volume, V_c , in accordance with Test Method D 2856 or equivalent test method.

9.1.3.3 Calculate the thickness of the damaged surface layer, TDSL, using the following relationship:

$$TDSL = (V_b - V_c)/A \tag{5}$$

Note 12—The volumes (V_b) and (V_c) and area (A) used in Eq 5 are determined from the Test Method D 2856 sample and *not* the thermal resistance sample.

9.1.3.4 Determine the effective diffusion thickness of the thermal resistance specimen, ΔX_{eff} .

$$\Delta X_{eff} = \Delta X_{geo} - 2 \times TDSL \tag{6}$$

9.2 Thermal Resistance Measurement:

9.2.1 Weigh the thermal resistance specimen to within 0.1 %. Measure the cross section and thickness of the specimen to within 0.1 mm (0.004 in.) and compute the specimen volume and apparent density.

9.2.2 Conduct the thermal resistance test under the desired temperature conditions in accordance with all of the requirements of the selected test method. At a minimum, tests shall be performed at a mean temperature of $24 \pm 2^{\circ}C$ ($75 \pm 4^{\circ}F$) with a temperature difference of 10 to $28^{\circ}C$ (18 to $50^{\circ}F$) imposed on the specimen. Additional conditions are allowed. See Notes 4 and 5.

9.2.2.1 When performing a series of tests on a specimen as a function of time, the specimens must be stored in an environment that replicates the mean temperature of the thermal resistance experiment when the specimen is not being tested. The means used to store the specimen shall not significantly interfere with the aging process. If the requirements of 9.2.2 are followed, the specimens shall be conditioned at $24 \pm 2^{\circ}$ C (75 $\pm 4^{\circ}$ F) and 50 ± 5 % relative humidity.

9.2.2.2 The TDSL will contribute to the total specimen thermal resistance. Therefore, it is recommended that all changes in thermal resistance be normalized as a percentage of the initial specimen thermal resistance measurement.

NOTE 13—The initial thermal resistance measurement is used to normalize the test data for standardization purposes. This measurement should be performed within 14 to 28 days after manufacture.

9.2.2.3 The normalized thermal resistance computed in 9.2.2.2 can be used to scale the initial thermal resistance of a

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full-thickness specimen based on the relationship detailed in 5.2.4. The performance of this initial full-thickness test is recommended. The thickness effect is the change in the thermal preformance of an insulation due to it's test thickness.(16)

NOTE 14—Since aging applies to the portion of the specimen that is closed celled, the scaling factor is based on effective diffusion thicknesses.

9.2.3 Because of the limited thickness of a single slice, a specimen comprised of several slices may be used to fabricate a thicker thermal resistance specimen. The user of this test method should ensure that the individual slices are stacked in a consistent manner for each test. The stacking reduces the systematic error due to the plate-to-specimen contact thermal resistance, but introduces contact thermal resistances between the layers and increases the overall amount of TDSL in the specimen.

9.2.3.1 Reducing the specimen thickness may alter the λ_r component due to the thickness effect. These concerns should be considered before initiating any series of experiments.

Note 15—If several thicknesses are scaled simultaneously, comparison of the results will address whether the λ_r component has been affected.

10. Report

10.1 Report the following information, including references to applicable test methods:

10.1.1 The name, address, and any other identification of the test laboratory and the date of the report.

10.1.2 The name and any other identification of the material tested, and the dates of testing.

10.1.3 The manufacturer of the material, the date obtained, and the date of manufacture.

10.1.4 The method of specimen preparation.

10.1.5 The geometric and effective diffusion thicknesses and the densities of the specimens. If an alternative to Test Method D 2856 was used to determine the TDSL, the procedure shall be fully described and its equivalency demonstrated.

10.1.6 The aging conditions.

10.1.7 The type and size of the thermal test apparatus.

10.1.8 The test mean temperature and temperature difference, and age of each specimen at each test time.

10.1.9 The apparent thermal conductivity and thermal resistance of each specimen at each test time.

10.1.10 If applicable, the time-averaged thermal resistance, selected service life, and the thermal resistance as a function of time.

10.1.11 A statement of the precision and bias of the test apparatus and procedure used to arrive at the reported data.

10.1.12 Any additional pertinent observations and remarks.

11. Precision and Bias

11.1 The precision of this test method is not known because interlaboratory data are not available. Interlaboratory data are being obtained, and a precision statement will be added with the next revision.

11.2 The precision of this test method is significantly influenced by the specimen preparation techniques and the dimension measurement procedures, as well as the precision of the thermal test method used. Precision data on these combined procedures are not yet available for all material types.

11.2 A round robin was conducted between 1993 and 1994, as reported by Graves, et. al. (17), and employed unfaced, rigid, closed-cell polyisocyanurate foam specimens from 7.6 to 33-mm (0.3 to 1.3-in.) thick. The polyisocyanurate boardstock was blown with hydrochlorofluorocarbon 141b and CO₂. Thirteen laboratores were involved in this study. The ratios of predicted lifetime thermal resistance to initial thermal resistance for the seven data sets on board Set 1 had a coefficient of variation of 1.1 % for 10-year lifetimes and 1.3 % for 20-year lifetimes for a thickness of 38 mm (1.5 in.) The ratios of predicted lifetime thermal resistance to initial thermal resistance for the eight data sets on board Set 2 had a coefficient of variation of 6.0 % for 10-year lifetimes and 6.6 % for 20-year lifetimes for a thickness of 38 mm (1.5 in.).

11.3 A round robin was conducted 1995, as reported by Fabian, et. al. (18), and employed a selected extruded polystyrene foam insulation. The specimens varied from 7.9 to 41 mm (0.31 to 1.6 in.) in thickness. Three laboratories participated in this study. The extruded polystyrene foam insulation used in the round robin was produced in January 1995 using HCFC-142 b as the blowing agent. The board stock was nominally 50 mm (2 in.) thick. Each laboratory prepared their own slices using different slice preparation methods including hot-wire, planer, slicer, and band saw. This comparison of slicing/scaling thermal resistance data for extruded polystyrene foamboard insulation resulted in a variability of no more than \pm 2.5 % in average thermal resistance computed for 38-mm (1.5-in.) thick specimens and \pm 2 % for 51-mm (2.0-in.) thick specimens. The 20-year time-averaged thermal resistivity values for specimens prepared using the slicer and the band saw agreed within 0.7 %. The 20-year time-averaged thermal resistivity values for specimens prepared using the hot wire were aobut 3 to 5 % higher than those of the band-saw and slicer specimens.

Note 16—Hot wires could produce surface skins that can affect the aging process as shown by the data available from Ref 18 when the hot wire is compared to the other preparation methods. This technique shall not be used as a specimen preparation technique.

<u>11.4 A</u> preliminary series of experiments compared the thermal resistance measurement portion of this test method. Four laboratories performing thermal resistance experiments at 24°C (75°F) on the same three test specimens of polyisocyanurate foam ranging in thickness from 32 to 64 mm (1.3 to 2.6 inches) reported an average deviation of 0.8 % and a 2σ (standard deviation) of 2.3 %. All of the participants utilized Test Method C 518 apparatus. The specimens used were approximately 25 days old (179).

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Note 167—The sliced thickness of the specimens in this comparison were significantly greater than those proposed by this test method. The effects of reduced thickness is under evaluation.

12. Keywords

12.1 aging; rigid closed_cell plastic foams; scaling factors; thermal insulation; thermal resistance; time-averaged thermal resistance

ANNEX

(Mandatory Information)

A1. SAMPLE CALCULATIONS

A1.1 Determination of Scaling Factor

A1.1.1 This calculation follows the procedure in 5.2.

A1.1.2 *Problem*—A 50-mm (2-in.) thick homogeneous rigid closed_cell plastic foam material is to be measured for its long term thermal resistance. An estimate of the thermal resistance at 3650 days (10 years) is needed.

A1.1.3 *Solution*—A 11-mm (0.43-in.) thick slice is removed from the center of a section of the insulation material following the guidelines described in Sections 7 and 8.

A1.1.4 Following the procedure detailed in 9.1.3, determine the TDSL using Eq 5 which, for this example, is assumed to be 0.25 mm (0.010 in.). The effective diffusion thickness is therefore 10.5 mm (0.41 in.).

A1.1.5 Using Eq 3, the thin-slice specimen shall be conditioned for 3650 days \times (0.0105 m)²/(0.050 m)² or 161 days in order to obtain the equivalent thermal performance of the full thickness section aged for 3650 days (10 years).

A1.1.6 The scaling factor is $(10.5/50)^2 = 0.044$.

A1.2 Estimation of the Time-Averaged Thermal Resistance

A1.2.1 This calculation follows the procedure in 5.3.1.

A1.2.2 *Problem*—The same material described in A1.1A1.1 has a service life of 3650 days (10 years). An estimate of the time-averaged thermal resistance is needed.

A1.2.3 Solution—Repeat the process described in A1.1.3 and A1.1.4 to prepare at least five test specimens and determine their effective diffusion thicknesses.

A1.2.4 Periodically measure the thermal resistance of each test specimen as a function of time for at least 161 days. It is recommended that at least ten measurements be performed on each specimen. Determine the relative (or normalized) thermal resistance of each specimen as a function of time by normalizing each specimen's test results to its initial measurement.

A1.2.5 Measure the initial thermal resistance of at least five full-thickness specimens. Using the average resistance as a baseline, calculate the thermal resistance of the full-thickness specimen as a function of scaled time using the average data obtained in A1.2.4.

A1.2.6 Curve-fit and integrate the data obtained in A1.2.4 to estimate the time-averaged thermal resistance for a service life of 3650 days (10 years). Report both the aging results as well as the time-averaged value.

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