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Title: Measuring the Impact of Experimental Parameters upon the Estimated Thermal
Conductivity of Closed-Cell Foam Insulation Subjected to an Accelerated Aging Protocol

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Abstract:

The thermal conductivity of many closed-cell foam insulation products changes over time as production gases diffuse out of the cell matrix and atmospheric gases diffuse into the cells. Thin slicing has been shown to be an effective means of accelerating this process in such a way as to produce meaningful results. Recent efforts to produce a more prescriptive version of the ASTM C 1303 standard test method have led to the initiation of a broad ruggedness test. This test includes the aging of full size insulation specimens for time periods up to five years for later comparison to the predicted results. Experimental parameters under investigation include: slice thickness, slice origin (at the surface or from the core of the slab), thin slice stack composition, product facings, original product thickness, product density, and product type. This paper will cover the structure of the ruggedness test and provide a glimpse of some early trends.

Introduction

Heat transfer through closed-cell foam insulation occurs via radiation, solid conduction, and gaseous conduction.[1] The radiation and solid conduction change little over time, but the gaseous conduction is determined by the composition of the gas mixture within the foam cells. Many closed-cell foam insulation products are produced such that the cells are initially filled with a gas that has a low thermal conductivity, relative to that of air. Over time, the cell contents change as atmospheric gases diffuse into the cells and as some of the initial gas(es) diffuse out into the surrounding environment. These changes in the molecular concentration, or partial pressure, of each of the cell gas components are governed by the diffusion coefficient for each gas, the foam thickness, and time.[2] For insulation sheets where the thickness is small relative to the width and length, this diffusion process has been shown to follow Fick's Law for one dimensional diffusion.[3-9]

Closed-cell foam insulation is used in buildings and appliances; applications with lifetimes ranging from eight to 40 years, or more. The energy efficiency of each application is directly related to the thermal resistance of the foam insulation. In the 1980s, appliance manufacturers and builders expressed interest in the long-term thermal resistance values, as opposed to the thermal resistance of new foam. Naturally, users would rather not wait 15 years to obtain these values, so accelerated aging methods were developed. Aging acceleration became especially important during the mid-1990s, when chlorofluorocarbons and hydrochlorofluorocarbons were ruled out as blowing agents for future products and it became necessary to evaluate the long-term thermal performance of candidate replacement blowing agents.

European age acceleration methods are based upon increasing the foam temperature because the gaseous diffusion coefficients are greater at elevated temperatures. However, because the relationship between the diffusion coefficient and temperature are different for each gas, there is

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no basis for comparing the age acceleration obtained by this method to the actual age of the foam. In contrast, age acceleration based upon decreasing the foam thickness can be directly correlated to actual foam aging via Fick's Law. Using this approach, cooperative research supported by the Polyisocyanurate Insulation Manufacturers Association and the Department of Energy during the late 1980s and the 1990s advanced both the specimen preparation techniques and the data analysis methodology.[10] An ASTM task group was formed during this time and the original version of the ASTM C 1303 test method was published in 1995.[11]

The flexibility within that test method, although desirable for research purposes, has been identified as a hindrance by industry stakeholders, who have noted that results can vary depending upon the interpretation, and therefore the implementation, of the version of ASTM C 1303 that was revised and published in 2000.[11] For example, the thin slice stacks employed by various users have included: (a) core slices only, (b) surface slices only, (c) cross sections of the whole product made up of 10 mm slices, and (d) cross sections of the whole product where the slice thickness was a function of the whole product thickness. No minimum slice thickness was specified in ASTM C 1303, which also contributed to the variability in results.

In 2000, a prescriptive test method based on ASTM C1303, but expanded to include permeably-faced products, was published in Canada (CAN/ULC-S770) and was required for products sold in Canada.[12] This extension of the methodology to permeably-faced products introduced more test practice variants. For example, there was confusion regarding whether a "surface" slice actually included the facer, or if the facer was stripped away before the slice was prepared. After considerable use of the Can/ULC-S770 procedure, it has been observed that there may be an inherent bias in the results and that the magnitude of the bias may be a function of the material tested and the slice thickness.

Spurred by these developments, and by the reluctance of the Federal Trade Commission to require the use of the more flexible ASTM C 1303, efforts began in 2003 to produce a prescriptive version and to expand the scope to include products with permeable facers. During the revision process, questions were raised regarding the applicability of accelerated aged performance values derived from measurements on 50 mm (2-in.) products to products of other thicknesses. (Cell morphology differences have been postulated to explain differences in aging behavior for products of differing thicknesses.) This latter issue is especially important considering that the prescriptive option within the most recent ASTM C 1303 may be used for product labeling purposes. There are also questions regarding the influence of variations within each material class, such as density, manufacturing process, and facer material.

As a first step in addressing these questions, a theoretical analysis of core and surface slice stacks was made to determine which type of slice would provide a more accurate acceleration of the aging process. However, considering the many product variations, empirical data was needed to ultimately determine which set of test method parameters would produce results most representative of the aged full thickness products.

A ruggedness test was therefore organized to answer the questions regarding product differences (class and thickness) and stack composition that were considered to be the most important.

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Another variable, slice thickness (in particular, the differences for slice thicknesses of 8, 10, and 12 mm), has been examined in a more limited fashion. The goals of the ruggedness test are to:

- Identify any difficulties or problems executing the new prescriptive method.
- Provide normalized data and statistical analysis sufficient to establish preliminary bias data for the test procedure (within the limitations of the single-lab condition).
- Examine relationships between product characteristics, stack composition, slice thickness, and the 5-year prediction bias via statistical data analysis.
- Examine the efficacy of the homogeneity and alternate product thickness qualification tests and to explore modifications to these test criteria if appropriate using statistical analysis of the test data.

Expanded polystyrene and polyisocyanurate manufacturers are participating in the first phase of this study. Later phases may include spray polyurethane, bun stock, or even pipe insulation products.

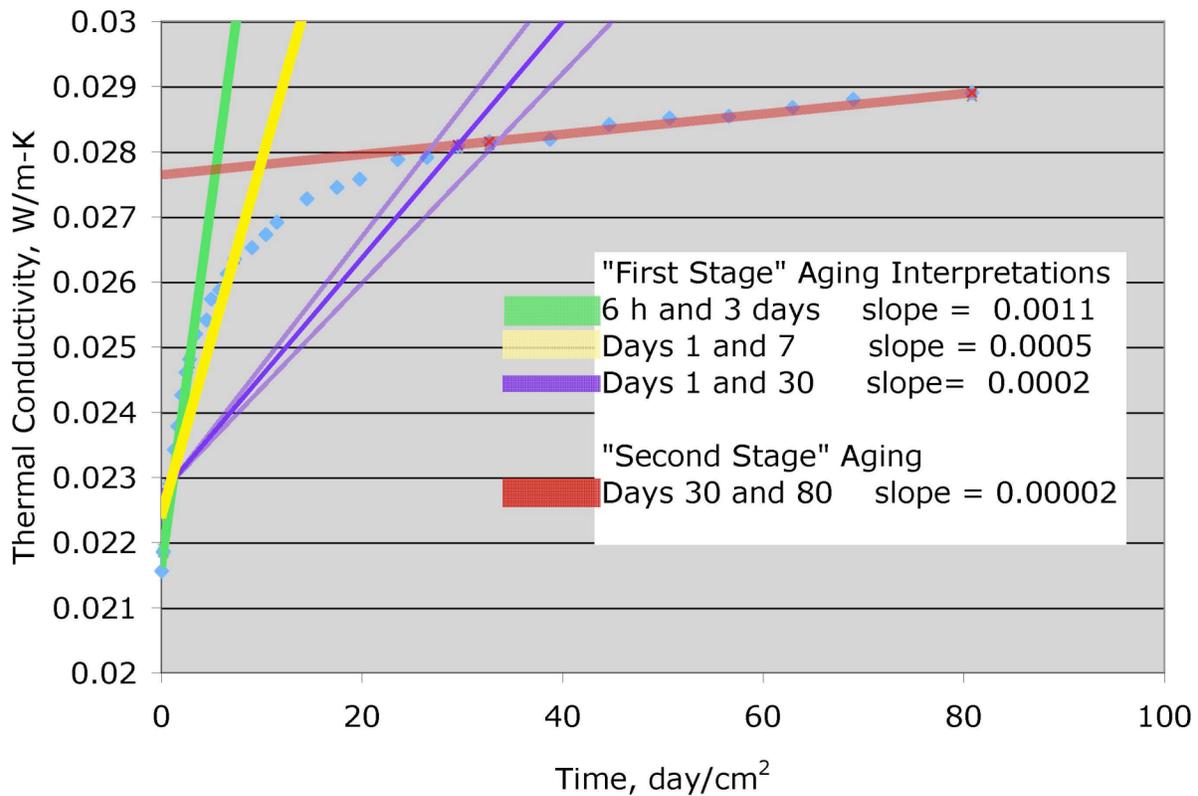
Theoretical Analysis of Core and Surface Slice Stacks

There have been differences of opinion regarding which type of slice, core or surface, best represents the full thickness product in the age acceleration process. As discussed above, the theoretical foundation for thin-slice age acceleration test method applies rigorously for homogenous foam. In real products, especially in faced-products, the surface foam region may provide additional gas diffusion resistance and therefore retard the aging process. There have also been questions regarding the degree to which portions of the foam cross section must be similar, or homogenous, for the age acceleration process to produce an acceptably accurate prediction of the full-thickness aged thermal conductivity.

A previous version of ASTM C 1303 defined a foam product as sufficiently homogenous if the slope of the thermal conductivity versus the normalized time during the first stage of aging didn't vary more than 10% between multiple specimens taken from the core and surface regions.¹ As shown in Fig. 1, this criteria left the definition of the 'first stage' of aging to the user.

¹ The normalized time was the time divided by the square of the slice thickness.

Figure 1 Homogeneity criteria from ASTM C 1303 (2000), showing the 10% acceptable bounds for an aging slope based upon data from days 1 and 30.



In revising the standard to produce a more prescriptive method, the method of examining the first stage of aging was altered into an “age equivalence” qualification criteria, shown in Eq. 1 based upon the ‘aging factor’ ratio approach from CAN/ULC S770.² In this qualification test, the change in thermal conductivity over a period of time of approximately one month for surface slices is compared to the corresponding change for core slices over the same time period. One of the objectives of the ruggedness test is to determine an appropriate ‘passing grade’ for this criteria. Until that information becomes available, the criteria have been arbitrarily set at a broad level.

$$\text{Age Equivalence} = 100\% \left[1 - \frac{2 \left[\left(\frac{k_{24h/cm^2}}{k_{30d/cm^2}} \right)_{\text{surface}} - \left(\frac{k_{24h/cm^2}}{k_{30d/cm^2}} \right)_{\text{core}} \right]}{\left[\left(\frac{k_{24h/cm^2}}{k_{30d/cm^2}} \right)_{\text{surface}} + \left(\frac{k_{24h/cm^2}}{k_{30d/cm^2}} \right)_{\text{core}} \right]} \right] \quad (1)$$

In order to understand the influence of homogeneity upon the predictive methodology, a simplified analysis was performed for a single polyurethane foam composition, and consisted of

² All variables are identified in a nomenclature list at the end.

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the following steps: (1) Determine the change in cell gas composition as a function of time and position. (2) Use the cell gas composition to determine the local time-varying thermal conductivity. (3) Apply this methodology to the appropriate geometries for core slices, surface slices, and full thickness specimens. (4) Compare the results for the modeled aging equivalence test found within ASTM C 1303 to the agreement between the modeled thin-slice predictions and the modeled full-thickness 5-year values.

Cell Gas Composition

In this analysis, the foam insulation was treated as a homogenous medium with constant effective diffusion coefficients. This is appropriate considering that the accelerated aging process takes place under controlled laboratory conditions with a relatively constant temperature. A two-zone model was used to reflect the greater diffusion resistance within a 'skin' layer at the surface. For purposes of gas diffusion, the skin was treated as a lumped diffusion resistance, that is, the gas contents within the skin itself were not calculated.

The geometries of interest include a surface slice, a core slice, and a full thickness slab. The core slice was modeled as continuous foam 10 mm (0.4 in.) thick. The surface slice was modeled with a total thickness of 11 mm (0.43 in.), consisting of 10 mm (0.4 in.) of core region and a 1 mm (0.04 in.)-thick skin. The gas diffusion resistance of the skin was varied as a multiple of the gas diffusion resistance of the core foam. This treatment is similar to that used in a previous combination of experiment and numerical modeling.[13] Full thickness slabs of 25, 50, and 75 mm (1, 2, and 3 in.) thickness were modeled, with and without two 1-mm (0.04 in.) skins included within that total dimension.

The gases used in this analysis were limited to nitrogen, oxygen, and isopentane; there was no attempt to model all gases used in all foam insulation products. An exact solution for the nitrogen, oxygen, and isopentane partial pressures within the foam continuum, as a function of time and location, was derived for each of the three geometries, as summarized in Table 1 and using the values shown in Table 2.

Table 1. Equations used to calculate the gas contents of the foam cells (based upon material from Hoogendoorn and Carslaw and Jaeger).[14-15]

Effective Diffusion Model: $\frac{\partial p}{\partial t} = D \frac{\partial^2 p}{\partial x^2}$		Initial Condition: $p = p_0$	
Nondimensional diffusion resistance ratio:	$H = \frac{LD_{skin}}{\delta D_{core}}$	Nondimensional time:	$\tau = \frac{D_{core} t}{L^2}$
Case one: unfaced foam (core slice or full thickness unfaced foam)			
Boundary Conditions	at $x=0$ and $x=L$; $p=p_{env}$		
General solution for a slab from 0 to L	$\frac{(p - p_{env})}{(p_0 - p_{env})} = 4 \sum_{n=0}^{\infty} \frac{1}{\beta_n e^{\beta_n^2 \tau}} \sin\left(\frac{\beta_n x}{L}\right)$ where $\beta_n = (2n + 1)\pi$ (For very small values of τ , up to 70 terms in the summation were required.)		
Case two: faced foam (full thickness faced foam)			
Boundary Conditions	at $x = 0, x = L$; $D_{core} \frac{\partial p}{\partial x} = \frac{D_{skin}}{\delta} [p(x, t) - p_{env}]$		
General solution for a slab from 0 to L	$\frac{(p - p_{env})}{(p_0 - p_{env})} = \sum_{n=1}^{\infty} \frac{2(\beta_n \sin \beta_n + H(1 - \cos \beta_n))}{\beta_n (\beta_n^2 + H^2 + 2H)} (\beta_n \cos \beta_n \frac{x}{L} + H \sin \beta_n \frac{x}{L}) e^{-\beta_n^2 \tau}$ the roots, β_n , are positive and satisfy: $\tan \beta_n = \frac{2\beta_n H}{\beta_n^2 - H^2}$ (Up to 80 terms of the summation were needed for very small values of τ .)		
Case three: foam faced on one side (surface slice)			
Boundary Conditions	At $x=L$: $p=p_{env}$ At $x=0$: $D_{core} \frac{\partial p}{\partial x} = \frac{D_{skin}}{\delta} [p(0, t) - p_{env}]$		
General solution for a slab from 0 to L	$\frac{(p - p_{env})}{(p_0 - p_{env})} = \sum_{n=1}^{\infty} \frac{2}{(\beta_n^2 + H^2 + H)} \left[\beta_n \cos \beta_n \frac{x}{L} + H \sin \beta_n \frac{x}{L} \right] \left[\sin \beta_n + \frac{H}{\beta_n} (1 - \cos \beta_n) \right] e^{-\beta_n^2 \tau}$ where: $\tan \beta_n = \frac{-\beta_n}{H}$ (Up to 19 terms in the summation were used.)		

Table 2. Gas Parameters

Gas	Viscosity (micro-Pascal-sec)(a)	Molecular Weight	Boiling Temp. (°C)	Thermal conductivity W/m-K (a)	Environmental pressure (atm)	Initial pressure in foam (atm)	Effective diffusion coefficient (cm ² /sec)
Nitrogen	17.9	28.02	-195.79	.02583	.79	0	2.35 E-8(b)
Oxygen	20.8	32	-182.96	.02658	.21	0	2.03 E-7(b)
Isopentane	6.7	72.15	27.9	.0149	0	.7	5.2 E-10(c)

Sources:
(a) CRC for 300K [16]
(b)Mitalas and Kumaran, Bhattacharjee, et al., and Biesmans, et al.[17-19]
(c) Singh et al, 2002 for 300K [13]

Total Thermal Conductivity:

The total thermal conductivity was modeled as the linear superposition of the thermal conductivity due to solid conduction, gaseous conduction, and radiation, as described in Glicksman.[20] The following assumptions were also based on material presented in that work for polyurethane foam:

- Gas was assumed to occupy 97% of the volume.
- The sum of the solid conductivity plus the radiation component was set = 0.009 W/m-K.
- The thermal conductivity of the 1-mm skin was set equal to 0.055 W/m-K or to the value of compressed polyurethane at 0.262 W/m-K for a ‘worst case’ non-homogeneity to examine the effect of this variable.
- The thermal conductivity of the gas mixture (λ_{mix}) was calculated using the Wassiljewa equation with the Lindsey and Bromley coefficients and the gas data shown in Table 2. [21,22]

Based upon these assumptions, the thermal conductivity at intervals of approximately 1 mm was calculated as shown in Eq. 2. These values were then used to determine the total thermal resistance of the slab as shown in Eq. 3.

$$\lambda(\text{location, time}) = 0.97\lambda_{mix}(\text{location, time}) + \lambda_{solid + radiation} \quad (2)$$

$$R_{total}(\text{time}) = \frac{\text{skin thickness}}{\lambda_{skin}} + \sum_{\text{core foam thickness}} \frac{\text{interval thickness}}{\lambda(\text{location, time})} \quad (3)$$

Analysis Results

The analysis results were used to examine the ASTM C 1303 “aging equivalence” homogeneity criteria for core and surface slices, per Eq. 1, and to compare the modeled 5-year predictions from the thin slices to the modeled full-thickness thermal conductivity at 5-years. Based on this two-zone model, the surface slices aged at a slower rate than the core slices, and the rate varied with the diffusion resistance multiple (DRM), as shown in Fig. 2. The surface slices display higher thermal conductivities in this figure because the thermal conductivity shown is that of the whole slice, which includes the 1-mm skin with a constant thermal conductivity, as shown in Fig. 3. The 1825 day (5 years) values for the full thickness products, predicted from the same analytical model, are superimposed on the thin slice results in both of these figures. All of the data shown in Fig. 2 are based upon a constant skin thermal conductivity of 0.055 W/m-K. This figure shows excellent agreement, as expected, between the core slices and the unfaced full thickness foam. However, for the non-homogenous skins modeled, neither the core nor surface slices produced a very close prediction of the full thickness aged thermal conductivity. The differences, or ‘error’, between the prediction and the full thickness value is shown numerically in Table 3. Another skin thermal conductivity of 0.262 W/m-K was also evaluated to provide a “worst-case” non-homogeneity condition, as shown in Table 3. However, even with this significant non-homogeneity, the “age equivalence” defined in Eq. 1 remained within a very small range, as shown in Table 3.

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Figure 2 Thermal conductivity for thin slices and full thickness of a hypothetical polyurethane foam from a two-zone model with a 1-mm skin thermal conductivity set to a constant value of 0.055 W/m-K.

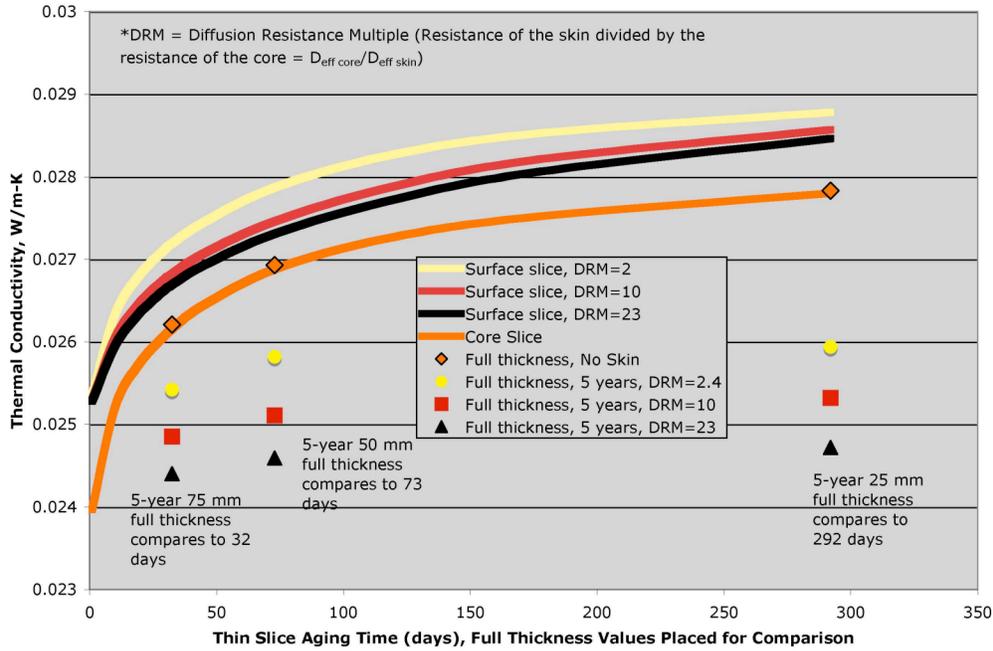


Figure 3 Thermal conductivity throughout the thickness of the 10 mm slice (skin 1 mm thick at $x/L=0$) and 50 mm full thickness pieces as calculated by the model for $D_{eff\ core}/D_{eff\ skin} = 10$.

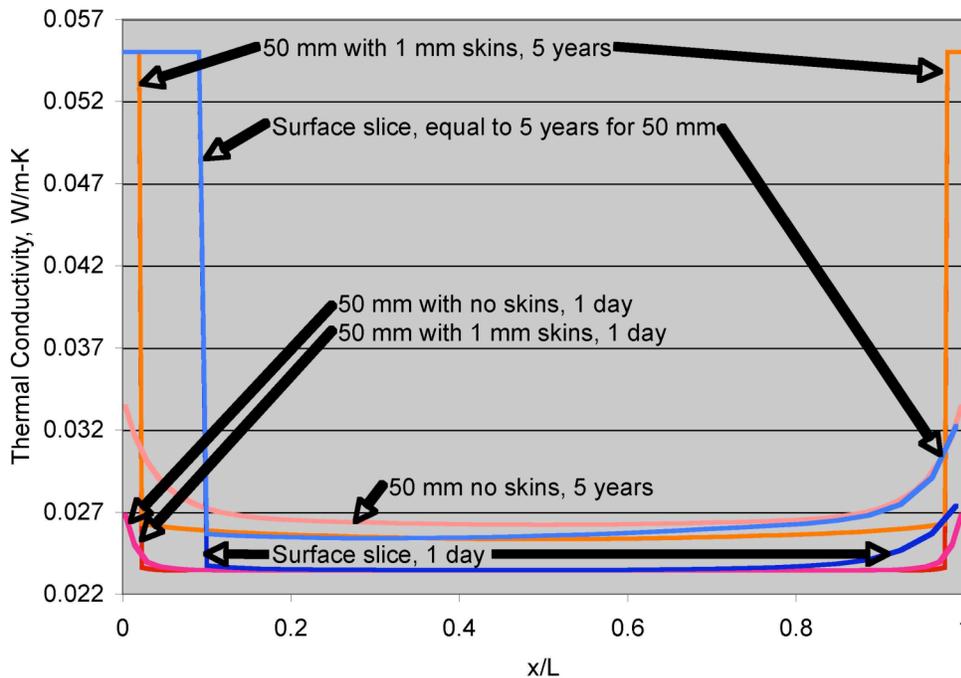


Table 3. Two-zone lumped diffusion resistance model results for core and surface slice aging predictions

1-mm skin thermal conductivity	$D_{\text{eff}}^{\text{core}}/D_{\text{eff}}^{\text{surface}}$	$\left(\frac{k_{24h/cm^2}}{k_{30d/cm^2}}\right)_{\text{surface}}$	$\left(\frac{k_{24h/cm^2}}{k_{30d/cm^2}}\right)_{\text{core}}$	Homogeneity Age Equivalence	Error range (%)*	
					Core	Surface
No Skin	1	1.09	1.09	100	0.1 to 0.3	
0.055	2	1.07	1.09	101.7		
0.055	10	1.06	1.09	102.8	5 to 10	8 to 13
0.055	23	1.05	1.09	103.2	7 to 12	9 to 15
0.262	2	1.07	1.09	101.5		
0.262	10	1.06	1.09	102.6	4 to 6	10 to 14
0.262	23	1.06	1.09	103.0	6 to 9	12 to 16

* Error = $100 \times \frac{k_{\text{slice prediction}} - k_{\text{full thickness at age}}}{k_{\text{full thickness at age}}}$; All values from theoretical model. The error range covers errors for 25 to 75 mm full thickness products.

Ruggedness Test Protocol

Considering the limited ability of any analysis to fully capture the product-specific property variations, a ruggedness test was organized to address the questions that were considered to be the most important. *A ruggedness test is designed to “test the test”, not to test the materials.*

The purpose of the ruggedness test protocol was to (1) identify and quantify the impact of sample preparation options, specifically thin slice stack composition, in application to multiple classes of polyisocyanurate foam and extruded foam products, (2) determine whether the aged performance predictions based on 50 mm (2-in.) products properly represent the aged performance of products of other thicknesses, also as a function of product class, and (3) provide guidance regarding the appropriate criteria for homogeneity.

The testing procedures included:

- Density- measured according to ASTM D 1622
- R-value of full thickness foam specimens measured after aging in a laboratory environment.
- R-value predictions per ASTM C 1303, including Homogeneity and Alternative Thickness Qualification tests
- Thickness of the destroyed surface layer was measured for a broad subset of the products.

Some of the variables in this study are inherent to the material and others are determined by the procedure. Both the product and test variables need to be studied, as outlined in Table 4. The product variables shown here were selected as representative of commercially available products. Each of the tests shown in this table will eventually be compared to a full thickness measurement.

Table 4 Variables Considered in Ruggedness Test

<u>Product Variables</u>			
Material Type	Manufacturers	Material Variations	Thickness (mm)
Polyisocyanurate	2	Type II, Class 1, Grade 1 (black facer)	25, 50, 100
		Type II, Class 2 (white facer)	25, 50
Extruded Polystyrene	2	Density: 27 kg/m ³ (1.7 lb/ft ³) (± 10%)	25, 50, 100
		Density: 48 kg/m ³ (3.0 lb/ft ³) (± 10%)	50, 100
<u>Test Variables</u>			
Slice Thickness	Product Variables	Stack Composition	
10 mm	All of the above	Core slices only	
		Surface slices only	
		2 Surface and 2 Core slices	
10 mm	100 mm thick products only	Full cross section	
8 and 12 mm	One 50 mm thick polyisocyanurate product	Core slices only	
		Surface slices only	
		2 Surface and 2 Core slices	
	One 50 mm thick extruded polystyrene product	Core slices only	
		Surface slices only	
		2 Surface and 2 Core slices	

Table 4 outlines a large number of variables encompassed within the ruggedness test. To the extent possible in any experiment, all other factors were held constant. The factors specifically addressed include:

- A single test laboratory performed all specimen preparation and thermal conductivity measurements.
- A single band saw was used for all thin slice preparation.
- A single ASTM C 518 measurement was prepared for each of the thin-slice combinations outlined in Table 4. (An initial investigation of the ASTM C 518 convergence criteria for these apparatus was made and the results were applied consistently for all thin slice measurements.)
- Multiple ASTM C 518 measurements were made and averaged for full thickness specimens whenever possible.
- All ASTM C 518 measurements made on one of three heat flux meter apparatus, regularly control-charted using the same stable specimen. This test environmental factor will be included in the statistical analysis.
- To the extent possible, all sample thicknesses for the same density (for XPS) and the same facers (for polyisocyanurate) were provided from the same plant location.

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- Samples were be provided in 1.2 x 1.2 m (4 x 4 ft.) pieces, except for some products that are only available in 0.6 x 1.7 m (2 x 8 ft.) boards.
- All thin-slice specimens were extracted in the same manner from the full thickness product sheets.
- All shuffling of thin slices between the core, surface, and mixed stacks took place in the same order for each product.

The following test factors were defined by ASTM C 1303:

- All samples were shipped between 7 and 12 days after the date of manufacture and all thin slice specimens were prepared between 14 and 21 days after the date of manufacture.
- All ASTM C 518 measurements were made at a mean temperature of 24°C (75°F) with a temperature difference of 22°C (40°F).
- All thin slices were prepared to meet the specifications outlined in C 1303, including flatness and agreement in average thickness for every slice within the stack.

The test schedule was designed to compare the predicted aged values, based upon thin-slice stack measurements, at time periods of 1, 2, 3, and 5 years for every product. The test schedule also includes measurements to test the ability of thin slices prepared from one product thickness to predict the 5-year thermal resistance of the “Alternate Product Thickness”. Time periods elapsed between specimen preparation and thermal conductivity measurements were calculated as shown in Eq. 4 (from ASTM C 1303) using the values summarized in Table 5. For the purpose of this calculation, the average slice thickness for surface slices does not include the thickness of any facing material.

$$\text{Test Time} = \left[\frac{\text{Average Slice Thickness}}{\text{Full Product Thickness}} \right]^2 \times \text{Time}_{\text{Full Thickness}} \quad (4)$$

Table 5. Test Schedule of ASTM C 518 Thermal Conductivity Measurements

Nominal product thickness used as specimen source (mm)	Specimen	“Full Product Thickness” used to calculate “Test Time” in Eq. 4* (mm)	“Time ^{full thickness} ” used to calculate “Test Time” in Eq. 4 (years)
25	Thin stacks	25	1, 2, 3, 5
		50 (AP)**	5
		100 (AP)	5
	Full thickness	Actual time	0.5, 1, 2, 3, 5
50	Thin stacks**	25 (AP)	2, 3, 5
		50	1, 2, 3, 5
		100 (AP)	2, 3, 5
	Full thickness	Actual time	0.5, 1, 2, 3, 5
100	Thin stacks	25 (AP)	5
		50 (AP)	5
		100	1, 2, 3, 5
	Full thickness	Actual time	0.5, 1, 2, 3, 5
25, 50, and 100	24-hour and 30-day tests as described in ASTM C 1303 Annex A1		
*The measured thickness of the full thickness product, as opposed to the nominal thickness, was used when available.			
**AP = Alternate product thickness comparison tests were only made for those cases where the full thickness specimen was included for comparison in Table 4. For example, Table 4 shows that only 25 and 50 mm product thicknesses are tested for Type II, Class 2 polyisocyanurate, so there is no need to prepare a prediction for a 100 mm product thickness for that category.			

Full thickness boards [nominal size 1.5 m² (16 ft²)] have been set aside to age undisturbed under standard conditions [22 ± 1 °C(72 ± 2 °F) and 40-60% RH] so that their thermal conductivity can be compared to the thin-slice aging predictions. At intervals shown in the test schedule in Table 5, a full thickness test specimen will be extracted from a full-size board, a minimum of 150 mm from the edge, and the R-value will be determined per ASTM C518.

For product boards that are 1.2 x 1.2 m (4 x 4 ft.), a maximum of four 300 mm (12 in.) squares can be harvested from each for thin slicing, and still remain at least 300 mm (12 in.) away from any edge. For product boards that are 0.6 x 2.4 m (2 x 8 ft.), more 300 mm (12 in.) squares can be harvested along the length of the board, but they are only 150 mm (6 in.) away from the edge. The number of boards needed from the manufacturers was estimated based on the more common size of 1.2 x 1.2 m (4 x 4 ft.), as summarized in Table 6. Each manufacturer has agreed to retain a sample of the product submitted for this study for the duration of the study (5-years).

Table 6. Insulation Boards Required

Product ID	Nominal product thickness (mm)	Number sections needed for thin slicing	Number of boards needed for thin slicing	Number of boards for full-thickness aging	Contingency	Total number boards for each product
Polyisocyanurate, White facer	25	6	2	5	2	9
	50	2	1	5	1	7
Polyisocyanurate, Black facer	25	6	2	5	2	9
	50	8**	2	5	3	10
	100	3*	1	5	2	8
XPS, 1.7 lb/ft ³	25	6	2	5	2	9
	50	8**	2	5	2	9
	100	3*	1	5	1	7
XPS, 3 lb/ft ³	50	2	1	5	1	7
	100	3*	1	5	1	7

* Two stacking configurations tested with the 100mm product

** 8, 10, and 12 mm slice thicknesses tested for one manufacturer only

Current Status of Ruggedness Test

As of June 4, 2007:

- >239 slices have been prepared.
- >1,900 slice thickness measurements have been made.
- ~300 mass measurements have been made.
- 100 Full thickness boards have been marked and stored.
- 675 ASTM C 518 thermal conductivity measurements have been made (85% complete).
- 26 full thickness specimens have been cut and measured (26% complete).
- A computer program has been written to automate the data extraction from the individual test files.
- The last test is scheduled for November 8, 2011
- Preliminary calculations for the homogeneity and alternate product thickness qualification tests have been made.

Preliminary summaries of the homogeneity and alternate product thickness qualification test results are shown in Tables 7 and 8. The homogeneity qualification test depends on a set of four ASTM C 518 test results to compare the aging behavior, over the first 30 days, of sets of slices taken from the surface and core of the material. The intent is to determine whether the foam is homogenous enough throughout its thickness so that a subset of that thickness, in the form of thin slices, can be used to adequately represent the aged thermal conductivity of the whole. Note that the values here all fall in a very narrow range of 94 to 97%. The usefulness of this test criteria, and a reasonable pass/fail criteria, won't be known until the full thickness aging is complete.

Table 7. Preliminary analysis results for the homogeneity qualification test requirements.

Material Type*	Material Variation*	Number of Sets**	$\frac{\lambda_{24h/cm^2}}{\lambda_{30d/cm^2}}$ <i>surface</i>	$\frac{\lambda_{24h/cm^2}}{\lambda_{30d/cm^2}}$ <i>core</i>	Average Homogeneity Age Equivalence (%)
All		18	0.94	0.91	96
A	all	8	0.95	0.90	95
A	x	5	0.96	0.90	94
A	y	3	0.94	0.92	97
B	all	10	0.94	0.91	97
B	x	6	0.94	0.92	97
B	y	4	0.93	0.90	97
Missing**		6			

*Material types and variations shown in Table 1. They are masked here by “A, B, x, y”.

**Each set consists of four ASTM C 518 thermal conductivity measurements. Those counted as missing were instances where at least one of the four ASTM C 518 tests failed to meet the test time requirements of the ASTM C 1303 prescriptive criteria.

The alternate product thickness criteria test is a bit broader. It seeks to determine whether the accelerated results from one product thickness can be used to predict the aged thermal performance of another product thickness. That is, can thin slices taken from a 50-mm (2-in.) thick product adequately predict the aged thermal conductivity of 25-mm or 100-mm (1-in. or 4 in.) thick products? Because of the comparative nature of the alternate product thickness qualification test, it requires a set of eight ASTM C 518 test results. These tests are used to compare the 30-day aging performance of core samples from one product thickness to core samples from another product thickness. A similar comparison is made for the surface slices. The values for these specimens range from 97 to 103% for the core stacks and 97 to 102% for the surface stacks. In addition to comparing the aging rates, the absolute thermal conductivities after 30 days of aging are compared for both core and surface sets from each product thickness. The values for these specimens ranged from 96 to 107% for the core stacks and 91 to 106% for the surface stacks. The preliminary results for eight such sets are shown in Table 8. Four other sets included in the original protocol failed to meet the test time criteria for at least one of the eight required ASTM C 518 tests. Again, the usefulness of these test criteria, and a reasonable pass/fail criteria, won't be known until the full thickness aging is complete.

Table 8. Alternate Product Thickness Qualification – Preliminary Results

Material Type*	Material Variation*	λ_{24h/cm^2}				Age Equivalence (%)		Thermal Conductivity Equivalence Core (%)	
		Core		Surface					
		50 mm	Other Thickness	50 mm	Other Thickness	Core	Surface	Core	Surface
A	x	0.900	0.907	0.970	0.961	101	99	96	91
A	x	0.900	0.875	0.970	0.946	97	98	103	103
A	y	0.901	0.929	0.945	0.943	103	100	102	105
B	x	0.916	0.941	0.944	0.950	103	101	96	98
B	x	0.916	0.902	0.944	0.941	98	100	102	102
B	x	0.907	0.931	0.933	0.950	103	102	99	99
B	y	0.919	0.915	0.937	0.941	100	100	101	100
B	y	0.889	0.870	0.930	0.901	98	97	107	106

*Material types and variations shown in Table 1. They are masked here by “A, B, x, y”.
 **Each set consists of eight ASTM C 518 thermal conductivity measurements. There are four sets, not included in this table, where at least one of the eight C518 tests failed to meet the test time requirements of the ASTM C 1303 prescriptive criteria.

Discussion

Based upon the preliminary two-zone theoretical analysis reported here, one would conclude that the “age equivalence” homogeneity test is a poor indicator of whether or not the age acceleration method will produce acceptably accurate results. In the model results, the calculated age equivalence values all fell within a very narrow band, despite the significant non-homogeneities included within the model. However, there are a number of limitations inherent in this analysis. Foam insulation morphology is a more continuous spectrum of density and property variations than can be represented with a two-zone model. Moreover, the analysis was only performed for polyurethane prepared with a single pentane; in practice, multiple pentane isomers and other gas mixtures are used. No attempt was made to model extruded polystyrene foam insulation morphology or chemistry.

These analysis limitations, along with other concerns, led to the establishment of the ruggedness test underway and reported here. Meaningful analysis must await the results of the full thickness thermal conductivity measurements that will be made about four years from now. We won’t know whether this calculation approach to homogeneity and alternate product thickness qualification test criteria will serve to adequately screen out materials or material variations where the age acceleration method does not adequately predict the full thickness aged values for several more years. The effects of the other test variables will also be analyzed at that time. It is even possible that we may learn that the screening process itself is not necessary. The data will also be used to explore the implementation of the research option within the test standard. In the future, the study may be expanded to include spray polyurethane, bun stock, or even pipe insulation products.

However, the experience has already produced some useful information. The homogeneity and alternate product thickness qualification tests depend on sets of ASTM C 518 test results to compare the aging behavior over the first 30 days and the thermal conductivity of different sets of thin slices. In the preliminary results reported here, there are a number of sets that are “missing” because at least one of the ASTM C 518 tests failed to meet the prescriptive test time requirement. To avoid such problems in the future, several changes to ASTM C 1303 have been made or are underway. First, equations were added to the ASTM C 1303 test procedure to clarify the calculation of test times. Second, the acceptable time limit on the 24 h test has been broadened from 0.5 to 1 h to facilitate the execution of the two (core and surface slice stacks) tests with a single HFMA. Third, an example has been prepared for this section of the test procedure to outline all the necessary calculations.

<u>Nomenclature:</u>	
D	Effective diffusion coefficient, cm ² /second
D _{core}	Effective diffusion coefficient for gas within the core foam, cm ² /second
D _{skin}	Effective diffusion coefficient for gas within the skin, cm ² /second
DRM	Diffusion Resistance Multiple = D _{core} /D _{skin} , dimensionless
H	Nondimensional number, analogous to a Biot number, representing the diffusion resistance of the core divided by the diffusion resistance of the skin
k _{24h/cm²}	Thermal conductivity after a period time equal to 24 h multiplied by the square of the slice thickness (in cm), W/m-K
k _{30d/cm²}	Thermal conductivity after a period time equal to 30 days multiplied by the square of the slice thickness (in cm), W/m-K
L	thickness of foam excluding the skin thickness, cm
p	partial pressure, Pa
p _{env}	partial pressure of the gas in the surrounding environment, Pa
p ₀	initial partial pressure, Pa
t	time
x	Dimension through the thickness of the slab, m
β	Equation root
δ	skin thickness, m
λ	Thermal conductivity, W/m-K
λ _{mix}	Thermal conductivity of the gas mixture, W/m-K
τ	Nondimensionalized time

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