Evaluation of Experimental Parameters in the Accelerated Aging of Closed-Cell Foam Insulation: Results after Five Years of Full-Thickness Aging

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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. Efforts to produce a more prescriptive version of the ASTM C1303 standard test method led to the ruggedness test (a "test the test" process) described here. This test program included the aging of full-size insulation specimens for time periods of five years for direct 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 provides a detailed evaluation of the impact of the test parameters on the accuracy of the five-year thermal conductivity prediction.

BACKGROUND

This study took place over a six-year time span and multiple papers have been published with intermediate results. The test protocol is described by Stovall and Bogdan (2007) and Stovall et al. (2012). The early material characterization results for the polyisocyanurate (PIR) and extruded polystyrene (XPS) are summarized by Stovall (2009) and Stovall et al. (2012).

Heat transfer through closed-cell foam insulation occurs via radiation, solid conduction, and gaseous conduction (Scheutz and Glicksman 1983). 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. This project focuses on the changes in thermal conductance due to the changes that occur over time as atmospheric gases diffuse into the cells and the blowing agent gas(es) diffuse out into the surrounding environment. These changes are governed by the diffusion coefficient for each gas for the cell wall polymer, the foam thickness, and time (Isberg 1988). 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 (Kumaran and Bomberg 1990; Bomberg 1990; Edgecombe 1989; Ball et al. 1978; Mullenkamp and Johnson 1983; Booth 1980; McElroy et al. 1991; Hoogendoorn 1994).

Appliance manufacturers and builders have expressed interest in the long-term thermal resistance values of existing foam, as opposed to the thermal resistance values of new foam, so accelerated aging methods were developed to test long-term thermal resistance values. An ASTM task group was formed and the original version of the ASTM C1303 test method was published in 1995 (Graves et al.1995; ASTM 1995). 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 foam insulation products sold in Canada (ULC 2000). An extensive interlaboratory comparison showed that the original CAN/ULC-S770 procedure produced biased results and that the magnitude of the bias varied according to the material tested and the slice thickness (Drouin 2009; Stovall et al. 2002). During the multiyear effort to improve C1303 (ma-

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jor updates were issued in 2000, 2007, and 2012), questions were raised by members of the ASTM C1303 task group at working group meetings. Many of these questions regarded the applicability of accelerated aged performance values derived from measurements on 50 mm (2 in.) products to products of other thicknesses. Within C1303 and in this paper, this is called an *alternate thickness* or *alternate product thickness* prediction. (Cell morphology differences have been postulated to explain differences in aging behavior for products of differing thicknesses.) There were also questions regarding the influence of variations within each material class, such as density, manufacturing process, and facer material. Also, since real foam insulation materials are not perfectly homogenous, slices taken from different locations in the cross section may exhibit different thermal conductivity and may age at different rates. Another question was whether or not adhered or intrinsic facings at the outer surfaces were more or less resistant to gas diffusion.

Two previous ruggedness tests had addressed questions regarding the cutting methods used to prepare the foam thin slices and the thickness of the destroyed surface layer that results from the cutting process (Fabian et al. 1997; Stovall 2007). The ruggedness test reported here was organized to answer questions regarding product differences (material, class or density, and thickness) and stack composition that were considered to be the most important for the 2007 prescriptive version of ASTM C1303. Another variable, slice thickness (in particular, the differences for slice thicknesses of 8, 10, and 12 mm [0.31, 0.39, and 0.47 in.]), was examined in a more limited fashion and was previously considered in the evaluation of CAN/ULC-S770 (Drouin 2009). This ruggedness test explored the applicability of accelerated aged performance values derived from measurements on one product thickness to products of other thicknesses for the same material and material class, i.e., alternate thickness or alternate product thickness predictions. In addition to the evaluation of experimental factor effects, this test was designed to provide an estimate of the test method accuracy. For this purpose, full-thickness uncut insulation specimens were aged within a conditioned laboratory space for five years so that the measured thermal conductivity at that point could be compared to the prediction produced via the accelerated test method. The full-thickness specimens were cut to fit within the test apparatus at the end of the aging period.

The thin slices were arranged in stacks for thermal conductivity measurements. This is done to ensure the thermal conductivity measurements are outside the regime of the "thickness effect," that is, to ensure the test specimen is optically thick (Glicksman 1994). The stacks differ in composition according to the origin of the slices, which may be taken from the core of the product or at the surface. For the PIR specimens, which were faced with permeable materials, the facers were included in the surface slices, but the slice thickness used for all calculations did not include the thickness of the facers. Five stacks were included in the ruggedness test:

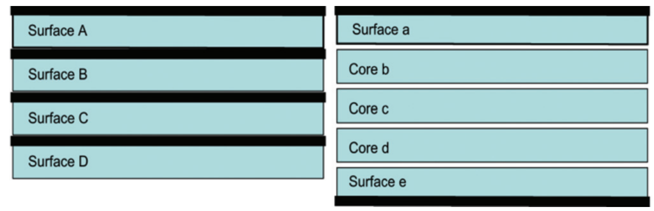

Figure 1 *Diagram of stack design: (left) Surface stack and (right) Profile Mixed stack.*

- The "Surface" stack is a simple stack of surface slices, all with surface side up, as shown on the left in Figure 1. The Surface stack includes the effect of any diffusion resistance provided by a densified surface or a facer, although each surface slice also includes an opposite cut surface without this extra diffusion resistance.
- The "Core" stack consists of only slices taken from the core of the product, that is, excluding the surfaces. The Core stack is composed of that portion of the foam that is relatively undisturbed by any diffusion that occurs between the time the foam is manufactured and when the thin slices are prepared.
- The "Profile Mixed" stack, shown on the right in Figure 1, is a replication of an actual cross-product section with two surface sides out and enough cores to make the stack height equal to the original product thickness, minus the thickness of the material destroyed during the slicing process.
- The "Four-Slice Mixed" stack consists of two outward-facing surface slices with two core slices in the center.
- The "Math" stack is a mathematical derivation, using the standard series resistance expression to weight the measured values from the Core and Surface stacks to represent the overall product structure, as described by Stovall et al. (2012).

During the course of this project, more than 1000 ASTM C518 thermal conductivity measurements were made (ASTM 2010). For the data used in the five-year comparison, there were 214 thermal conductivity measurements for slice stacks and 100 for full-thickness specimens. The thermal conductivity of all 20 full-thickness products was measured. All the thermal conductivity measurements were made on one of four heat flux meter apparatus. For most materials, there were at least three full thickness measurements made after five years of aging. For all full thickness materials, at least one measurement was made using a 600 × 600 mm (24 × 24 in.) specimen in a Fox 605 heat flux meter apparatus with a 20 × 20 mm (8 × 8 in.) metering area. For 25 and 50 mm (1 and 2 in.) thick specimens, that test specimen was then cut into four equal pieces so that multiple measurements could also be made in a Fox 304 heat flux meter apparatus with a 10 × 10 mm (4 × 4 in.) metering area. That same Fox 304 machine was used for most of the thin slice measurements; a Fox 300 machine was used for the remaining thin slice measurements.

ANALYSIS

In order to produce a robust statistical analysis, every individual prediction was compared to every corresponding full-thickness measurement. That is, the value predicted for five years from each of the five thin-slice stacks was compared to each of the replicate full-thickness measurements individually. In the cases where replicate measurements were available for the thin-slice stacks, each predicted value was individually compared to the full-thickness five-year measurement. The comparisons include predictions made using original specimens from both the same product thickness and from other product thicknesses. The resulting data set therefore reflects unbalanced multiple effects. The final data set contains a family of multiple predicted values for each measured full-thickness value, resulting in a total of ~1300 observations. The error, or difference between the predicted value and the measured value as defined in Equation 1, was calculated for each observation.

$$\text{Error (\%)} = \frac{\left(k_{Predicted} - k_{Full\ Thickness}\right)}{k_{Full\ Thickness}} \times 100\% \qquad (1)$$

where k is the thermal conductivity, W/m·K.

Using this definition:

- A positive error indicates the predicted thermal conductivity was too high—and therefore the predicted thermal resistance was too low.
- A negative error indicates the predicted thermal conductivity was too low—and therefore the predicted thermal resistance was too high.

The data set also included corresponding values for multiple possible effect classes. Some of these classes are associated with possible variations in the test methodology, such as stack type, whether or not predictions can be made using products of alternate thickness, or slice thickness. Other classes are useful in examining whether the test method accuracy varies between different applications, that is, whether it works equally well for different material varieties or products from different manufacturers. A few variables were considered as continuous numerical values rather than as classes of distinct values. A partial listing of the classes included in the data set are summarized in Table 1. Screening classes for homogeneity and alternate product thickness were examined as described by Stovall et al. (2012).

Statistical analysis was used to compare the response, or dependent variable, to the independent model variables. General linear models were used to identify which experimental factors were most important. The population marginal means (PMM; also called *LSMeans* in SAS [2007]) were used to determine the influence of each parameter in the model (SAS 2007). The PMM is a function of both the structure of the parametric model and the data (Searle et al. 1980). For example, the PMM for the effect of a Core stack will be different depending on which other effects were included in the model. If the model looked at the effects of stack type (Surface, Core, Mixed, Profile, and Math) and product variation (Class1 and Class 2), the PMM for Core stack effect would be the average of independent averages for Core stacks in Class 1 and Core stacks in Class 2. If the data set were perfectly balanced, there would be an equal number of test data points in each of those averages and the overall mean for Core stacks would equal the PMM for Core stacks. However, in an unbalanced data set, there will be more data points in one class than in the other. In that case, the PMM serves to balance an unbalanced data set. Note that the PMM can only be calculated if there is at least one observation for each possible combination of the effects in the model.

Table 1. Experimental Parameter Classes Used in Analysis

Classes	Possible Values
	Methodology Classes
Product thickness	25, 50, 100 mm (1, 2, 3 in.); note 75 mm (3 in.) included in 100 mm (4 in.) class
Original product thickness	Applies when alternates product thickness is used to make the prediction
Stack type	Surface, Core, Four-Slice Mixed, Profile Mixed, and Math
Machine comparison	Same or Different, whether the heat flux meter apparatus used to measure the full-thickness specimen was the same as the one used to measure the thin-slice stack that produced the predicted value
Thickness comparison	Same or Alternate, whether the original specimen used to produce the prediction was of the same thickness as the full-thickness specimen
Slice thickness	8, 10, or 12 mm (0.31, 0.39, or 0.47 in.)
	Application Classes
Variety	Class 1 or Class 2 for PIR, Low or High density for XPS
Manufacturer	Four included, two each for PIR and XPS

The analysis also produces the F-statistics, the explained variance divided by the unexplained variance as shown in Equation 2, which is useful in determining which experimental parameters are most important within the test protocol. The Type III ANOVA used here is unbiased, that is, the results are independent of the order in which the class variables are considered (Gill 2001; Pasta 2011). Also, the Bonferroni t-test form was used to adjust for the large number of parameters in the model (Miller 1981). This form of t-test provides a more rigorous test of significance for the influence of each parameter in the model.

$$F = \frac{\text{explained variance}}{\text{unexplained variance}} \tag{2}$$

RESULTS

The raw data are summarized in Figures 2 to 4. Figure 2 shows that the bulk of the predictions are within ±5% of the full thickness values, but there are also some outliers. In Figure 3, the same data set is broken down further to show the impact of stack type and whether predictions are based upon slices taken from the same product thickness or an alternate product thickness. Looking at both of these figures, it is apparent that some sort of screening is needed for the alternate thickness predictions. Further, it appears that Surface stacks would not be a good choice for PIR and that Core stacks would not be the best choice for XPS.

The test plan called for most of the test slices to be 10 mm (0.39 in.) thick, with some at 8 mm (0.31 in.) and others at 12 mm (0.47 in.) to evaluate the impact of that test parameter. Previous unpublished work done during the development of CAN/ULC S770 has shown that it is difficult to maintain the necessary slice flatness, that is, the uniformity of the slice thickness, for slice thicknesses smaller than 8 mm (0.31 in.) (Drouin 2012). Figure 4 shows that the actual execution represents a more continuous spectrum of slice thickness. Therefore, statistical models were explored to consider slice thickness both as a continuous numerical value and as a class variable with three levels.

The PIR and XPS were treated separately for the general linear model analyses, as were the alternate product thickness predictions and the same product thickness predictions. Before conducting the linear model analysis, the distributions of the results for these four groupings of interest were evaluated as shown in Figure 2. The distributions are sufficiently close to a normal distribution to justify the linear model approach. The final models had correlation coefficients of 0.84 and 0.86 for alternate thickness comparisons and 0.55 and 0.63 for same thickness comparisons, for PIR and XPS, respectively.

The F-statistics (see Equation 2) indicate which experimental parameters (see Table 1) are more closely correlated with the experimental error. The original product thickness, i.e., the thickness of the product used to produce the thin slices, appeared in every statistically significant correlation. The manufacturer variable showed little to no effect on its own, but for alternate product thickness comparisons the combination of manufacturer and product thickness was important. For PIR, the product variety (Class 1 or Class 2) was not correlated with the experimental error. However, the XPS product variety (standard or high density) was influential for same thickness predictions. Some variables were confounded by their close association with other variables. For example, the test duration for all 75–100 mm (3–4 in.) products was less than 40 days, so those two factors were ineffective when placed in the same model.

Two experimental factors were of special interest: stack type and slice thickness. Stack type plays a critical role in test method accuracy for both of these two materials. Slice thicknesses between 8 and 12 mm (0.31 and 0.47 in.) were statis-

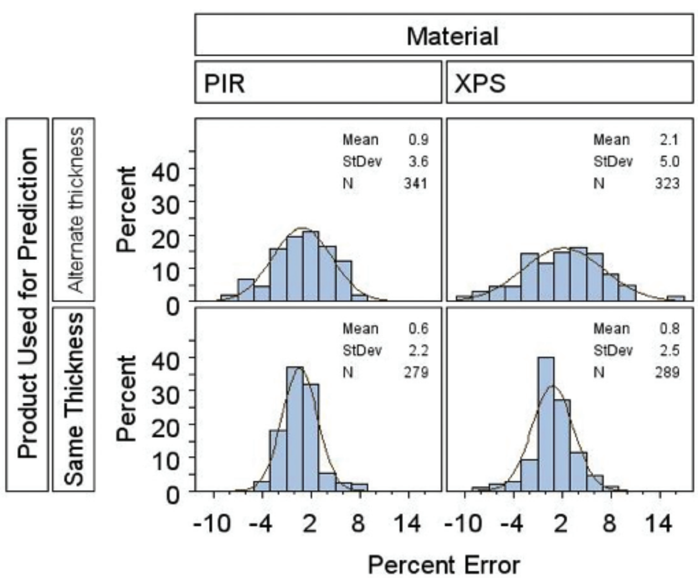

Figure 2 *Unscreened data set, range of values for PIR and XPS for alternate thickness comparisons and same thickness comparisons.*

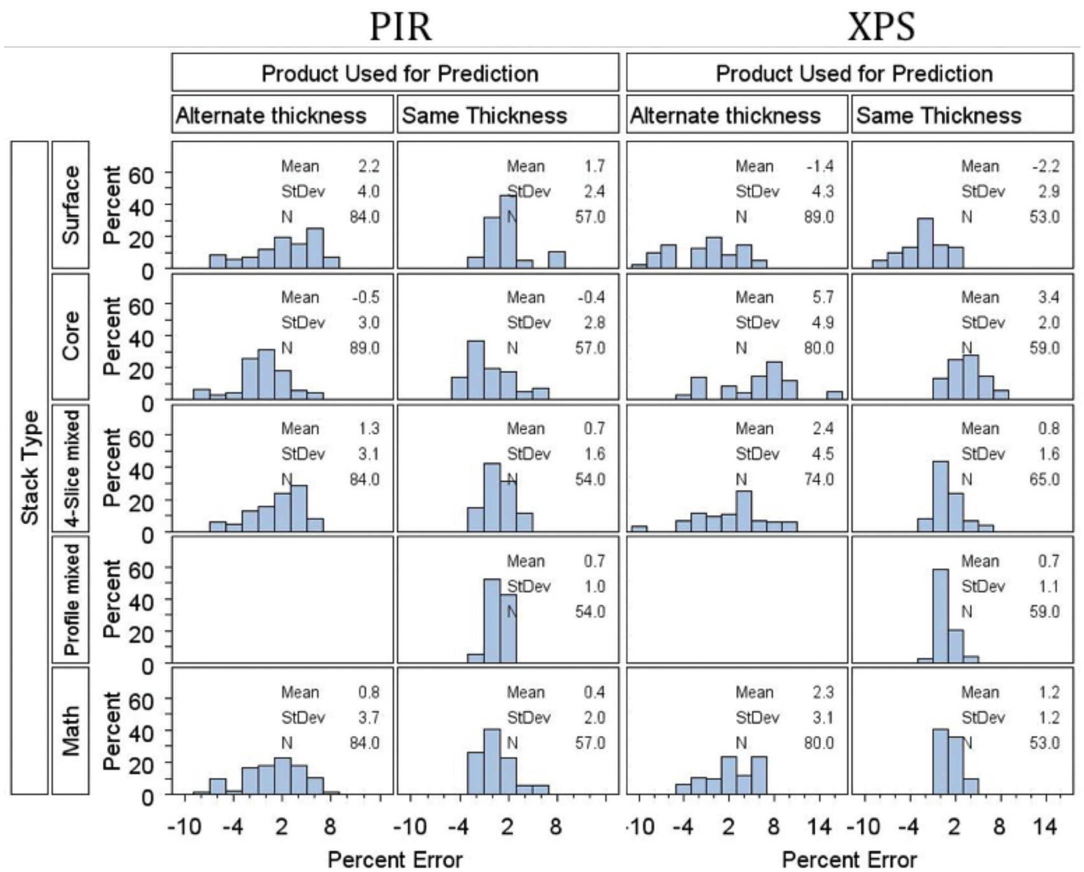

Figure 3 *Unscreened data set, range of values for PIR and XPS for both comparisons (same and alternate thickness) for five stack types.*

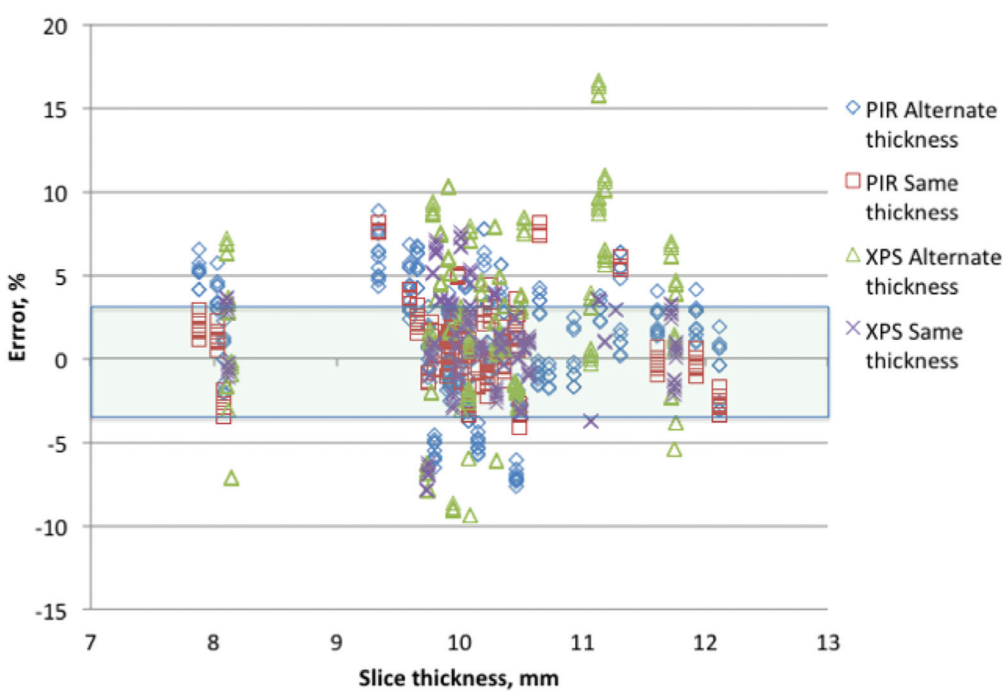

Figure 4 *Unscreened data set, range of values for both materials and both comparisons as a function of average slice thickness within the test stack.*

tically significant only for PIR predictions made from the same product thickness.

PIR, Alternate and Same Thickness Predictions

The PMM values for PIR, shown in Table 2, indicate that (1) the Core and Math stacks produce more accurate results; (2) using 100 mm (4 in.) products produces large errors, even when used to make predictions for products of that same thickness; and (3) results for slice thickness, significant only for same thickness predictions, were the same for 8 and 10 mm (0.31 and 0.39 in.) slices and much worse for 12 mm (0.47 in.) slice thickness, which is contrary to expectations based upon the relative magnitude of the errors introduced by the thickness of the destroyed surface layer (TDSL) (Stovall et al. 2012).

While the PMM values correct for the unbalanced nature of the data set, the means and standard deviations for the actual test values support these conclusions, except that the results for 12 mm (0.47 in.) slices appear to be very nearly the same as for 10 mm (0.39 in.) slices. Also, the Profile Mixed stack produced good results for the same thickness prediction.

Using these trends to guide the selection of test methodology options by deleting the Surface stack and the predictions based on the original product thickness of 100 mm (4 in.), the means for subsets of the actual test values are shown in Table 3. Considering the means and standard deviations, the most robust approach to the test methodology would appear to be to use Core stacks from 50 mm (2 in.) products to produce alternate thickness predictions and to use Profile Mixed stacks to produce same thickness predictions. The Math stack also offers reasonable results for same thickness and for alternate thickness if combined with the alternate thickness qualification criteria.

Several application factors were also statistically significant in the general linear models. For alternate thickness predictions, the combination of product thickness and manufacturer was the most important factor in the model, based on the Type III F-values as discussed above. Similarly, the combination of product thickness and variety were the third most important factor for same thickness predictions. The impact of the selected test methodology options for these application factors are shown in Table 4.

XPS, Alternate and Same Thickness Predictions

The PMM values in Table 2 for XPS indicate the following.

- The most accurate stacks for same thickness predictions are the two Mixed stacks and the Math stack.
- The most accurate stacks for alternate thickness predictions are the Surface and Math stacks.
- Using 75–100 mm (3–4 in.) products produces large errors for alternate thickness predictions.

Table 2. Multi-Factor Analysis Results: Population Marginal Means (PMM) of "Error"*

Class	Class Value	Alternate Thickness		Same Thickness	
		PIR	XPS	PIR	XPS
Stack	Surface	2.2	–1.0	1.6	–2.2
	Core	–0.8	5.5	–0.5	3.4
	Four-Slice Mixed	1.5	2.4	0.9	0.8
	Profile Mixed	NA	NA	0.9	0.7
	Math	0.9	2.1	0.3 (89% CL)	1.1
Slice origin product thickness	25 mm (1 in.)	–1.0	Not sig.		
	50 mm (2 in.)	0.6	0.8		
	75–100 mm (3–4 in.)	3.5	4.9		
Product thickness, Class 1 for PIR and standard density for XPS (other variations not significant)	25 mm (1 in.)			–2.1	2.4
	50 mm (2 in.)			2.4	0.7
	75–100 mm (3–4 in.)			5.0	2.3
Nominal slice thickness	8 mm (0.31 in.)			1.3	
	10 mm (0.39 in.)			1.2	
	12 mm (0.47 in.)			–3.9	

* Greater than 99% confidence level (CL) except where indicated otherwise.

Table 3. PIR Data Set Multiple-Effect Results for Error (%) for Test Methodology and Screening Criteria Classes

Original Product Thickness, mm (in.)	Stack Type	Alternate Thickness Criteria B*	Alternate Thickness			Same Thickness		
			Error	Std. Dev.	No. of Comparisons	Mean	Std. Dev.	No. of Comparisons
25 (1)	Core	Pass	0.1	2.6	10	–0.1	2.4	23
		Fail	–7.0	0.5	9			
	Four-Slice Mixed	Pass	1.0	2.2	13	0.4	2.0	23
		Fail	–5.0	0.6	9			
	Profile Mixed			NA**		0.6	1.1	23
	Math	Pass	–0.3	2.6	10	0.5	1.3	23
		Fail	–6.6	0.5	9			
50 (2)	Core	Pass	–0.6	1.6	41	–1.1	2.7	28
		Fail	–0.3	0.9	6			
	Four-Slice Mixed	Pass	1.6	2.5	38			
		Fail	2.6	0.9	6			
	Profile Mixed			NA**		0.6	0.9	28
	Math	Pass	1.0	2.6	41	–0.2	1.6	28
		Fail	4.0	0.9	6			

*Alternate thickness criteria B is based on taking the average of the thermal conductivity comparisons (core to core and surface to surface) at 30 days, with a 95% to 105% passing criteria. See Stovall et al. (2012).
**Profile Mixed stack is not applicable when slices from one product thickness are used to predict the aged thermal conductivity for products of another thickness.

- The same thickness predictions for 50 mm (2 in.) products were more accurate than for other product thicknesses.

While the PMM values correct for the unbalanced nature of the data set, the means and standard deviations for the actual test values support these conclusions. Looking at the actual test values, it would appear that the best stack selections would be the Math stack for alternate thickness predictions and either the Profile Mixed or Math stack for same thickness predictions. For alternate thickness predictions, it appears that only 50 mm (2 in.) products should be used.

Several application factors were also statistically significant in the general linear models. For alternate thickness predictions, product thickness was the most important factor in the model, and the combination of product thickness and manufacturer was the fourth most important, based on the Type III F-values. Similarly, variety (standard or high density) was the second most important, and the combination of product thickness and variety were the third most important factor for same thickness predictions. Table 5 shows how the recommended test method options of Math and Profile Mixed stacks perform for these application categories.

Data Distributions for PIR and XPS Boardstock for Selected Test Method Options

In addition to looking at the PMM and the data set means and standard deviations, histograms are useful in examining whether the results are sufficiently clustered near small error values. Core stacks were found useful for PIR products, while Math stacks were found useful for XPS products. For same thickness predictions, Profile Mixed stacks worked well for both products.

Figure 5 shows the relevant data distributions for these test method options. Looking at the top two quadrants of this figure, remember that the alternate product thickness method is only recommended when the slice origin product thickness is 50 mm (2 in.). The bottom right quadrant of Figure 5 shows the data distribution when the Math stack is used to produce predictions for both boardstock products using a slice origin product thickness of 50 mm (2 in.). Another accelerated aging protocol, CAN/ULC S770, also uses the Math stack approach but is based on an aging factor that is then applied to the initial full-thickness value, so the error values reported here will not be applicable to that test method (ULC 2000).

DISCUSSION

Test method recommendations for the two products included in this ruggedness test were presented based on both

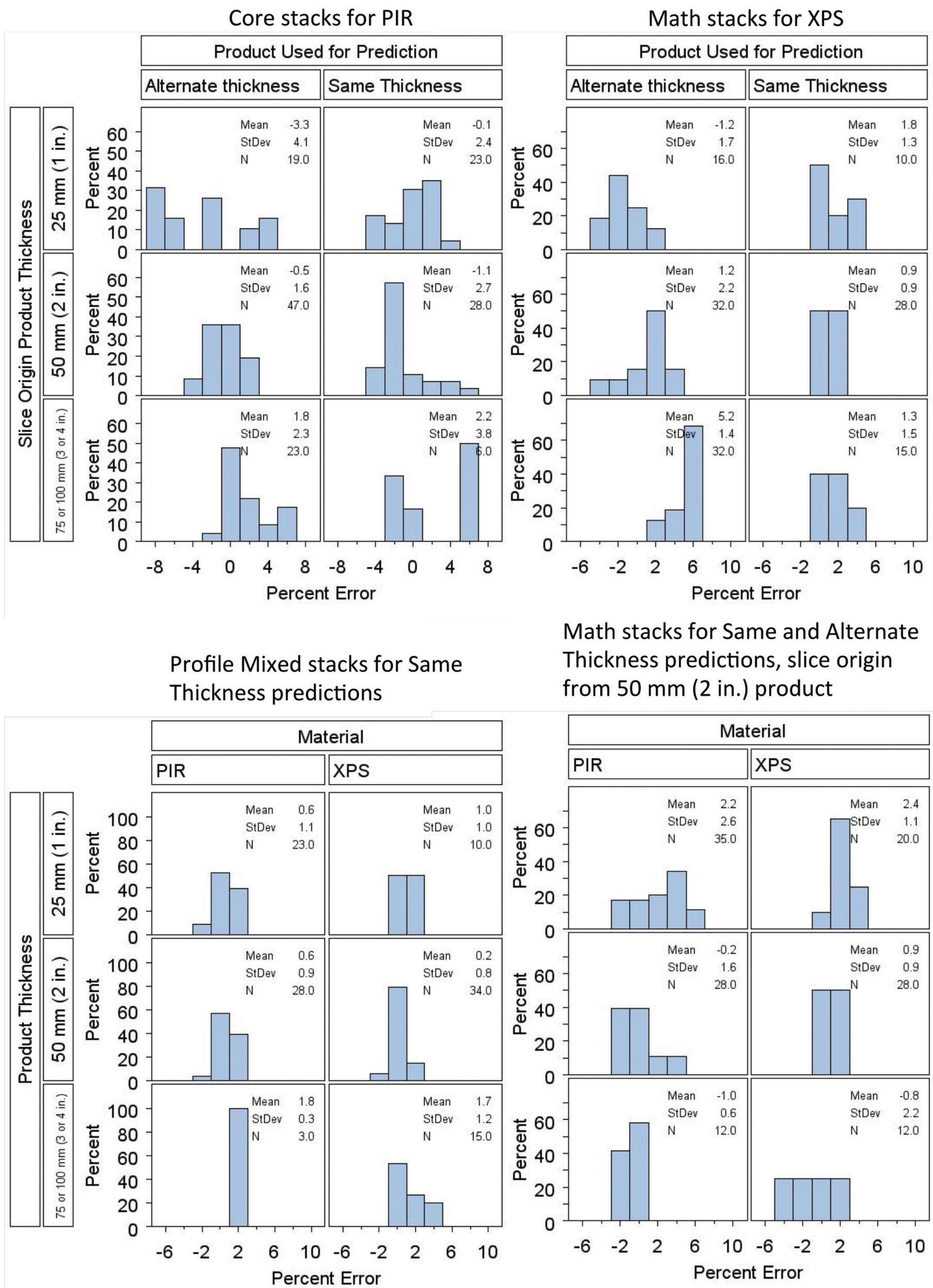

Figure 5 *Measured experimental error when selected stacks are used to produce both same and alternative thickness predictions.*

Table 4. Evaluating Applications for Selected Test Methodology Options for PIR Boardstock

	Product Thickness, mm (in.)	Mean of Error	Standard Deviation of Error	Number of Comparisons
*Alternate thickness prediction using **Core** stacks from **50 mm (2 in.)** original product thicknesses*				
Manufacturer a	25 (1)	0.7	1.1	23
	100 (4)	–2.6	0.6	9
Manufacturer b	25 (1)	–1.3	0.6	12
	100 (4)	–1.0	0.4	3
*Alternate thickness prediction using **Math** stacks from **50 mm (2 in.)** original product thicknesses, including only those that pass Alternate Thickness Criteria B**				
	25 (1)	1.8	2.7	29
	100 (4)	–1.0	0.6	12
*Same thickness comparison using **Core** stack type*				
Class 1	25 (1)	–0.6	3.0	12
	50 (2)	–2.7	0.5	18
	100 (4)	2.2	3.8	6
Class 2	25 (1)	0.6	1.2	11
	50 (2)	1.6	2.7	10
*Same thickness comparison using **Profile Mixed** stack type*				
Class 1	25 (1)	–0.2	0.9	12
	50 (2)	0.5	0.9	18
	100 (4)	1.8	0.3	3
Class 2	25 (1)	1.5	0.7	11
	50 (2)	0.8	1.0	10
*Same thickness comparison using **Math** stack type for 25–50 mm (1–2 in.) products*				
	25 (1)	0.5	1.3	23
	50 (2)	–0.2	1.6	28

*Alternate thickness criteria B is based on taking the average of the thermal conductivity comparisons (core to core and surface to surface) at 30 days, with a 95% to 105% passing criteria.

Table 5. Evaluating Applications for Selected Test Methodology Options for XPS Boardstock

	Product Thickness, mm (in.)	Mean of Error	Standard Deviation of Error	Number of Comparisons
*Alternate thickness prediction using **Math** stack type from 50 mm (2 in.) original product thicknesses*				
Manufacturer c	25 (1)	4.2	0.0	5
	75–100 (3–4)	1.2	0.8	6
Manufacturer d	25 (1)	1.8	0.5	15
	75–100 (3–4)	–2.8	0.3	6
*Same thickness comparison using **Profile Mixed** stack type*				
Standard Density	25 (1)	1.0	1.0	10
	50 (2)	0.4	0.8	22
	75–100 (3–4)	3.1	0.2	6
Higher Density	50 (2)	–0.2	0.6	12
	75–100 (3–4)	0.8	0.2	9
*Same thickness comparison using **Math** stack type*				
Standard Density	25 (1)	1.8	1.3	10
	50 (2)	1.1	0.7	16
	75–100 (3–4)	2.7	0.6	6
Higher Density	50 (2)	0.5	0.9	12
	75–100 (3–4)	0.4	1.0	9

the population marginal mean analysis (see Table 2) and the raw data analysis (see the error distributions in Figure 5). These recommendations are summarized in Table 6. Some recommendations are common to both materials:

- The Profile Mixed stack is excellent for same thickness predictions.
- The Math stack is acceptable for alternate thickness predictions.
- Don't use slices from 75–100 mm (3–4 in.) products for alternate thickness predictions—slices taken from a 50 mm (2 in.) product gave the best results.

The current edition of C1303 allows the use of the three test stack configurations as replicates. As the stack configuration is refined based on this ruggedness test, the issue of sufficient replicate measurements is obviously important and must be addressed by the task group.

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Table 6. Summary of Recommendations for Both Product Types Included in the Ruggedness Test

	PIR	XPS
Same thickness	Math or Profile mixed stack except for 100 mm (4 in.) products Core stack except for 100 mm (4 in.) products only	Profile mixed stack Math stack
Alternate thickness	Math stacks from 50 mm (2 in.) products that pass the alternate thickness criteria Core stacks from 50 mm (2 in.) products.	Math stack from 50 mm (2 in.) products. Surface stack from 50 mm (2 in.) products for 25 mm (1 in.) products

preparation is critical to this test method, and Jerry Atchley accomplished most of that work with assistance from Dr. Thomas Petrie.

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