# Accelerated Aging Test Methods for Predicting the Long Term Thermal Resistance of Closed-Cell Foam Insulation

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**ABSTRACT**

The accurate estimation of the thermal performance of insulation products used in buildings over their expected lifetime has been a recognized challenge for over 25 years. This is because the lifetime of such products is long, thermal aging is caused by the diffusion of a multitude of gases, and the insulation product is not homogeneous. The task of developing a standard test method for predicting long term thermal performance which applies to a variety of closed-cell foam products is even more complicated as diffusion processes occur at rates that depend on the type of polymer, the cellular structure, the temperature, the gas type, and its pressure. Both classical approaches to accelerating diffusion controlled phenomena, namely aging at higher temperature and aging a thin slice, present challenges especially if a single method is desired for a variety of cellular foam products such as polyisocyanurate (polyiso) and extruded polystyrene (XPS).

Though Europe has favored standard test methods based on aging at elevated temperature, slicing and scaling techniques have been the leading approach in North America. Lately, two prescriptive test methods, ASTM C 1303 in the USA and CAN/ULC-S770 in Canada have emerged in North America. Both are based on accelerating the foam aging process by slicing the foam into thin specimens. Both methods use the projected thermal conductivity at five years of age to represent the insulation's long term thermal resistance value (LTTR). The two methods have many other similarities, such as use of Fickian law for one-dimensional diffusion to calculate aging period, and use of a thin slice from the core and surface areas of the foam. However, they do vary in precisely how the long term thermal resistance is calculated. The C 1303 test method prescribes that the thermal resistance value of a stack of thin slices after aging for a calculated time is the long term thermal resistance value. The S770 test method calls for multiplication of an aging factor to an initial thermal resistance value to determine the five-year value. Though the basic frameworks of the two methods are in place, the precise parameters are still being debated and balloted.

This paper compares the two methods, ASTM C 1303-07 and CAN/ULC-S770-03 for their suitability for use as standard test methods by the polyiso and XPS insulation industry for their boardstock products. Mathematical modeling and calculation algorithms developed by Huntsman and described in earlier papers are used along with published thermal aging data to evaluate how effective the two methods will be to meet the various criteria for being an industrially useful method. It looks at the impact on the bias for each of the method for the various parameters still being debated, such as slice thickness and stack composition. This study demonstrates that with the appropriate choice of test conditions, each of the two methods have potential to give low bias with polyiso boards. For XPS boards, ASTM C 1303-07 appears to be the only real choice.

**INTRODUCTION**

Heating and cooling of buildings represents a large fraction of the total energy used by mankind. With the increased cost of fuel and concerns about global warming, there is an increased focus on improving the thermal performance of the building system. Decisions about insulation usage are among the most important an architect or specifier will make relative to the operational cost and environmental impact of a building. To achieve an overall building thermal performance, architects and specifiers know the thermal resistance (R-value) needs for a particular building, component, or component

system. They need the manufacturer's of building insulation products to supply them with reliable products with high, proven R-values.

Among all the insulation products, only polyisocyanurate and extruded polystyrene foam boards are closed celled plastic foams that are designed to capture a gas with much lower thermal conductivity than air. This makes such plastic foam insulation the most efficient insulator among all widely used commercial products. It is the reason why it has the highest growth rate among all insulations globally (1). Due to other requirements on such cellular foam insulations, especially when used in commercial roofing applications, they are not encapsulated in air-tight barriers. This causes the gas composition in the foam to change with time as air diffuses into the foam and the insulating gases diffuse out. The diffusion of some of the gas components is rather slow, leading to gradual shift in R-value over years to even centuries, stopping only when equilibrium concentrations are reached for every gas.

This presents a challenge. Certainly architects and specifiers want products that are made using the most advanced materials and process technologies while meeting all the current and anticipated environmental requirements. At the same time, they want to accurately represent the thermal performance. This makes it necessary that there be a relatively short duration, easy to implement and yet technically correct method to measure the thermal performance of closed celled plastics foams for the duration of its use in a building system. As elaborated in a 2002 publication by Huntsman, a prescriptive test method, CAN/ULC S770-00 (hence forth referred as S770) developed in 2000, is exactly such a method for the current polyisocyanurate boards used in commercial roofing (2).

**S770 Test Method**

S770 test method is based on acceleration of aging by slicing and scaling (3). It was developed under the auspices of the Underwriters' Laboratories of Canada (ULC) by a task group comprising of representatives from National Research Council of Canada (NRC) and all sectors of the plastic foam industry. It advocates that thermal performance of cellular foam insulation should be defined as the time-weighted average of R-value over 15 years at room temperature at the used thickness. This is primarily because the average useful life of a typical roof on a commercial building is 15 years. Field aging data, though limited, has confirmed that laboratory aging at room temperature adequately represents aging on a roof with all the temperature/humidity variations through the years (4, 5). For all of this paper, room temperature is understood to be a temperature of $22^oC \pm 5^oC$ ($72^oF \pm 9^oF$) and a relative humidity of 50% ± 20% and R-value measurement is understood to be done according the ASTM C 518 or C 177 with an average test temperature of $24^oC \pm 2^oC$ ($75^oF \pm 4^oF$) with a temperature difference of $22^oC \pm 2^oC$ ($40^oF \pm 4^oF$). It has been mathematically derived that for long term typical R-value aging (i.e., an exponential decay of R-value over years), the time-weighted average over 15 years is equivalent to the actual R-value after approximately 5 years (6). The S770 test method uses a slicing and scaling based methodology to predict this 5 year aged R-value in weeks to months. The S770 standard and other publications (3, 6) describe the exact procedure in detail but it entails the following three steps:

(a) Determination of the mean initial thermal resistance of the product, $R_{Product,\ initial}$

(b) Determination of the aging factor as the ratio between the thermal resistivity of a 6-12 mm thick slice at the specified time of aging ($R_{Slice,\ aged}$) to its initial value ($R_{Slice,\ initial}$). The specified time of aging is found using the formula

$$\text{Time of aging in days} = 5 \times 365 \left[ \text{Thickness of slice} / \text{Thickness of full thickness product} \right]^2 \quad (1)$$

The traditional approach to the slicing method has been to cut the thin slice from the core of the cellular board, but the S770 method requires cutting slices of the same thickness from both the surface and core. This is done to account for the long standing position that cellular insulations are not homogeneous in the thickness direction. Thus two aging factors, one for the core layer and another for the surface are determined.

(c) LTTR is calculated using the larger of the two aging factors, surface or core. Thus,

$$LTTR_{S770} = \frac{R_{Product,\ initial} \times R_{Slice,\ aged}}{R_{Slice,\ initial}} \quad (2)$$

The S770 method states that the initial thermal resistance of the product, $R_{Product,\ initial}$ shall be determined within 7-14 days after the production date and all the thin slices must be prepared on the same day as the measurement of initial R-value of the product. In addition, the method states that initial R-value of the slices ($R_{Slice,\ initial}$) must be measured within 2 hours of cutting the slice as the thin slices age very rapidly. There are other conditions such as if slices are stacked to measure R-value, the core stack cannot contain surface slices or vice-versa; during aging, both surfaces of the slices should be exposed to the ambient air; slice thickness should be uniform and so on. The test is considered invalid if the two aging factors, surface and core, are different by more than 12%. In this case the product is too heterogeneous and slicing and scaling principles do not apply to the product. A 2003 revision of the S770 method, CAN/ULC-S770-03, put an additional requirement that $R_{Slice,\ initial}$ can not be more than 12% lower than $R_{Product,\ initial}$ and that $R_{Product,\ initial}$ shall now be determined within 3-14 days after the production date.

CAN/ULC-S770 is incorporated by reference in the 2001 and subsequent editions of the Canadian standards for polystyrene, polyisocyanurate and spray polyurethane foam insulation: CAN/ULC-S701, "Standard for Thermal Insulation, Polystyrene, Boards and Pipe Covering:" CAN/ULC-S704, "Standard for Thermal Insulation, Polyurethane and Polyisocyanurate, Boards, Faced:" and CAN/ULC-S705.1, "Standard for Thermal Insulation – Spray Applied Rigid Polyurethane Foam, Medium Density Material-Specification." In the U.S., the S770 methodology was incorporated into the 2002 and subsequent editions of ASTM C 1289, "Standard Specification for Faced Rigid Cellular Polyisocyanurate Thermal Insulation Board."

As evidenced from some recent publications, comparison of results from S770 test data with actual aged R-value measured on full thickness product for polyiso boards and comparison of S770 data with historical aged R-value data for XPS boards suggest that the LTTR predicted by the S770 method is higher than the actual/historical aged data, i.e., there is a positive bias (8, 9). A study conducted under the auspices of Polyisocyanurate Insulation Manufacturers Association (PIMA) suggest that for the set of polyiso boards used in the study, the average bias is approximately +6% (8). Various XPS manufacturers have reported an over-prediction of 10-25% in their product literature. The ULC Thermal Insulation Committee has recognized the limitations of the current test method and has reconvened a task group to address the issue of bias in the next revision. In the mean time, the ASTM C1303 task group has approved a new test method ASTM C 1303-07, "Standard Test Method for Predicting Long-Term Thermal Resistance of Closed-Cell Foam Insulation" which includes a prescriptive method to complement the long-standing research method (10).

**ASTM C 1303 Test method**

Though many research papers had been published on the use of slicing and scaling to predict aged R-value of specific cellular plastic foams, ASTM C1303-95 was the first standard test method for estimating the long-term thermal resistance of unfaced rigid closed-cell plastic foam. Though this test method did not mandate a specific time period, it provided a methodology to calculate time-weighted average R-value for any period. Validation of this method came from a 5 year project at Oak Ridge National Laboratory (ORNL) in Oak Ridge, TN that showed good correlation among in-place polyiso roof insulation R-values with those measured on products stored in a laboratory and those predicted from laboratory slicing and scaling (4). Surprisingly this test method was not adopted as a method to specify LTTR values in any of the closed celled foam insulation standards. Though the exact reason stated by each type of material polyiso and XPS, is somewhat different, a common complaint was that it is too complicated to perform and requires a large number of measurements. A revision of the test method published as ASTM C 1303-00 in year 2000 included some simplifications but still neither the polyiso nor the XPS material standards adopted it (11). In 2003, the ASTM C 1303 task group decided to work towards adding a prescriptive element to the test method and ASTM C 1303-07 is a result of that work (10).

Similar to S770, the prescriptive part of the ASTM C 1303-07 (henceforth referred as C1303) defines LTTR as R-value at an age of five year which corresponds to the average thermal resistance over a 15-year service life (Ref 4, 5). Here, LTTR is defined simply as

$$LTTR_{C1330} = R_{Slice,\ aged} \quad (3)$$

Where $R_{Slice,\ aged}$ is the thermal resistivity of a 9 mm or higher thickness slice after aging for a time period defined by Equation (2). The method requires that slices be prepared between 7-14 days after the production date. At present C1303 requires that $R_{Slice,\ aged}$ data be collected using a stack of core slices only, a stack of surface slices only and a mixed stack of slices representing a cross-section of the product. Stacks of slices are used for thermal resistance measurements in order to minimize errors associated with radiation heat transfer phenomena between the hot and cold plates of the measuring device when separated by small specimen thicknesses. The method outlines many other requirements such as the foam portion of

each surface slice be a minimum of 9 mm; slices be uniform in thickness; slicing technique be geared towards minimization of damaged layers of cells; both surfaces of slice be exposed to free air circulation during aging; there be no air gap between slices during R-value measurement of the stack; stack orientation be consistent and so on. The standard method should be consulted for detail on each of these.

Recognizing that neither polyiso nor XPS are perfectly homogeneous, C1303 method sets forth a homogeneity qualification to insure that they are homogeneous enough for this test method to produce meaningful results. Aging characteristics of a stack of core thin slices is compared to a stack of surface thin slice specimens using the following formula:

$$\text{Aging Equivalence} = 100\,\% \left\{ 1 - \frac{2\left[ (k_1 / k_2)_{\text{Core}} - (k_1 / k_2)_{\text{Surface}} \right]}{\left[ (k_1 / k_2)_{\text{Core}} + (k_1 / k_2)_{\text{Surface}} \right]} \right\} \qquad (4)$$

Where $k_1$ is the thermal conductivity of a stack after aging for (24 ± 0.5) hours per (cm of slice thickness)$^2$ from the moment the initial cut was made on the full thickness product to make the thin slices and $k_2$ is the same after aging for (30 ± 1) days per (cm of slice thickness)$^2$. Thus if the slice thickness is 9 mm, $k_1$ and $k_2$ should be measured after (24 x $0.9^2$ = 19.44) hours and (30 x $0.9^2$ = 24.3) days of aging respectively from the moment the initial cut was made on the full thickness product to make the thin slices.

Currently, the C1303 method stipulates that if the aging equivalence is between 90% and 110%, the insulation specimen satisfies the homogeneity qualification for the use of the test. If not, accelerated aging results may not be used. The standard also has an alternate product thickness qualification which defines when test results for specimen taken from a particular product thickness shall be considered representative of other product thicknesses more than 1.3 cm (0.5") apart and made from identical components and having identical foam morphology. Alternate product thickness qualification is not in the scope of this paper.

The research part of ASTM C 1303-07 provides a relationship between thermal conductivity, age and product thickness. The calculation methods can be used to predict the R-value at any specific point in time as well as the average R-value over a specific time period. Many of the requirements of the prescriptive procedure, such as age of the product at the start of testing, homogeneity qualification, slice thickness, stack composition, aging environment and so on are only a guideline for research portion of the ASTM C 1303-07 method.

Though there appears to be willingness to adopt the C1303 test method by the polyiso and XPS industry, they are awaiting results of a ruggedness test currently underway at ORNL. The ruggedness test examines the influence of several test variables, most importantly thin slice stack composition, slice thickness, and product homogeneity on the bias with room temperature aging of full thickness product. The five year bias results will be available in the year 2011.

**BASIC ISSUES**

The above discussions and a review of the wider literature on the aging of closed cell foam suggests that there has been a tremendous effort by universities, national research laboratories, board insulation industry groups, and test standard bodies to first understand the topic of heat transfer and thermal aging and then to deduce a test method for the purposes of product evaluation, specifications or product comparison (12, 13). At the same time, it is clear that the only test available in USA and Canada at present, the S770 method, has a bias and a proposed new method is in the early stages of validation. Keep in mind, in most cases, 5-year actual aging is not an option due to continuing need to change the product composition and the cellular morphological characteristics to meet the various environment requirements (ozone depletion, green house gas, recycle content and so on) and to benefit from advances in polymer science.

Other approaches, namely aging at higher temperature and numerical modeling have been considered and deemed less desirable. The primary limitation of this method has been that a specific increase in temperature does not equally change the diffusion coefficients of all the gases involved in the aging process and whether the elevated temperature could damage the cellular structure of the foam. Given that LTTR is now being defined as the R-value at a specific time, 5 years, it is likely that at least for some types of cellular foam blown with a specific blowing agent, one can find a temperature which overcomes both limitations. After all, acceleration of aging by raising temperature is favored by standard making bodies in many European countries (14).

Several numerical models of the gas diffusion process through foam with location dependent diffusion coefficients have been proposed and each have the capability to accurately predict aged R-value of a given polyiso or XPS boards with essentially no bias (15-17). The difficulty with the numerical modeling is the effort required to measure all the input parameters. As an example, Distributed Parameter Continuum (DIPAC) model requires input of over 25 parameters including density, initial blowing agent fraction and polymer index for each blowing agents, effective diffusion coefficients at two temperatures for $N_2$, $O_2$, and each of the blowing agents; and all for core layer and surface layer (15). This is very onerous and unlike any standard test method for any material.

This suggests that the two prescriptive methods, S770 and C1303, are the best hope for practical LTTR approaches to be widely used by industry. In this paper we examine the reasons behind the high bias in the S770 method for XPS boards and the smaller bias in polyiso boards and steps that can be taken to reduce both significantly. An objective for this work is to ascertain what has the potential to give a lower bias across the range of polyiso and XPS products. We do all this by using the thermal aging simulation software developed by Huntsman Polyurethanes called Agesim to predict the thermal aging curves for thin slices and full thickness products using the input parameters obtained from published literature for both XPS and polyiso boards (2). This allows us to look at the effect on bias in LTTR of various parameters such as age of the foam, slice thickness, and time since slicing for initial R-value measurements, very expeditiously compared to experimentation and in some cases in ways not possible experimentally.

## THERMAL AGING SIMULATIONS: AGESIM

Huntsman's thermal aging simulation software, Agesim, is described in detail in an earlier publication and should be consulted for a fuller description (2). For each foam specimen, it requires conditions of aging; dimension, temperature of aging and R-value measurements. It also requires input of cell gas partial pressures and effective diffusion coefficients at the temperature of aging for each of the gases involved, namely $N_2$ and $O_2$ (or air), and the blowing agent(s). In order to simulate the aging behavior of products containing a densified skin layer and/or a surface density gradient having different diffusion characteristics than core foam, a one mm thick skin layer with effective diffusion coefficient, $D_{eff}$, defined as

$$D_{eff} \text{ through skin/facer} = X * D_{eff} \text{ through core} \qquad (5)$$

"X" is referred to as the skin factor and is calculated iteratively by fitting measured k-factor aging curves of the laminate with those predicted by Agesim. The solid conduction and the radiative heat transfer components of thermal conductivity are assumed constant in Agesim and all aging is attributed to changes in gas phase conduction due to diffusion of gases.

Huntsman has been using the Agesim software for a number of years to accurately predict R-value aging curves for polyiso boards. We have built an extensive database of diffusion coefficients, skin factors, and initial cell gas partial pressures through various foam laminates. As an example, Figure 1 shows the modeled and measured thermal resistance for a laminate board produced in 1998. This is a reproduction of Figure 6 from Reference 2 with additional measured data gathered since 2002. The measured diffusion coefficients and skin factors are listed in Table 3 of reference 2 and re-listed in the Table in section "calculations on polyiso boards" here.

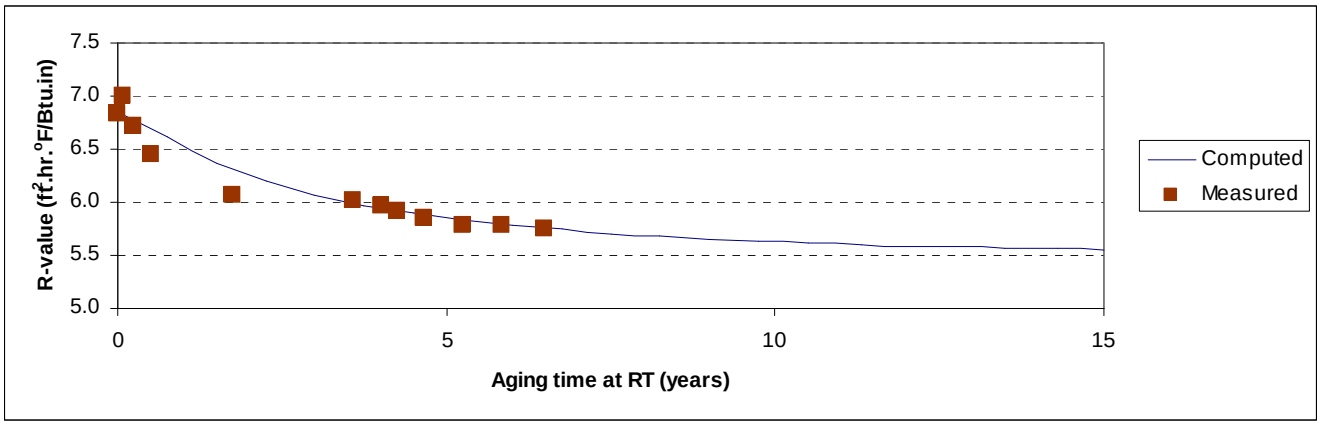


Figure 1: Predicted and measured R-value for a polyiso board

## CALCULATIONS ON XPS BOARD

Input data for XPS boards were found in published literature. Many sets of such data along with various aging curves exist. Given that the XPS boards presently being marketed in USA and Canada are blown using HCFC-142b alone, we focused primarily on $D_{eff}$ of $N_2$, $O_2$ (or air), and HCFC-142b. In 2010, XPS boards will have to start using a zero ozone depletion potential blowing agent in USA. A review of literature suggested that XPS is likely to use a mixture of two blowing agents, one with $D_{eff}$ similar to HCFC-142b and another with much higher $D_{eff}$ like that of HCFC-22. We came across more than a dozen sets of $D_{eff}$ data for $N_2$, $O_2$ (or air), HCFC-142b and HCFC-22 and the three sets we used in the illustrations given in this paper are shown in Table 1. XPS #1 & #2 are representative of current products and XPS#3 is representative (for the reason given above) of future products. The following specific Figures and Tables in the listed references were most influential in determining $D_{eff}$ and other material parameters listed in Table 1:

XPS # 1: Reference 18, a 1997 paper by B. Fabian et. al. – Table 1-6
XPS # 2: Reference 19, a 2004 paper by C. Vo – Table 3 and Figure 11
XPS # 3: Reference 20, a 2001 paper by C. Bratt et. al. – Table 3 and Figure 5

**Table 1: Input parameters used to model various XPS foam boards**

| Gas | Effective Diffusion Coefficient, $D_{eff}$ ($10^{-12}$ $m^2s^{-1}$) at RT | | |
|---|---|---|---|
| | XPS # 1 | XPS # 2 | XPS #3 |
| Nitrogen | 55 | 140 | - |
| Oxygen | 106 | 270 | - |
| Air | - | - | 50 |
| HCFC-142b | 0.75 | 0.059 | 0.2 |
| HCFC-22 | - | - | 20 |
| **Other Parameters** | | | |
| Density, pcf (kg/m$^3$) | 1.6 (25.6) | 2 (32) | 2 (32) |
| Thickness, inch (mm) | 2 (50.7) | 2 (50) | 2 (50) |
| Skin Factor | 1/5 | 1 | 1/2 |

Figures 2 and 3 show the Agesim predicted R-values for a XPS board and a skin slice cut from it along with the measured valued listed in reference 18. This is the quality of fit demanded in all cases before settling on the parameters listed in Table 1 and the other input parameters used in the model. The cell gas pressures for fresh board were taken as 0.008, 0.002 and 0.7 bars of $N_2$, $O_2$ and HCFC-142b respectively for XPS #1 and #2. For XPS #3, initial cell gas pressures were taken as 0.01, 0.39 and 0.31 bars for air, HCFC-142b and HCFC-22 respectively. A skin factor of 0.2 for XPS #1 means that it contained a 1 mm thick diffusion retarding skin on both surfaces having $D_{eff}$ of 1/5th of the core foam $D_{eff}$ for each gas.

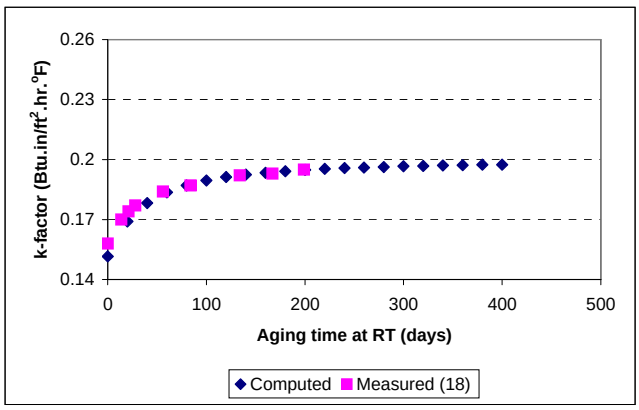



Figure 2. Computed and measured k-factor of laminate

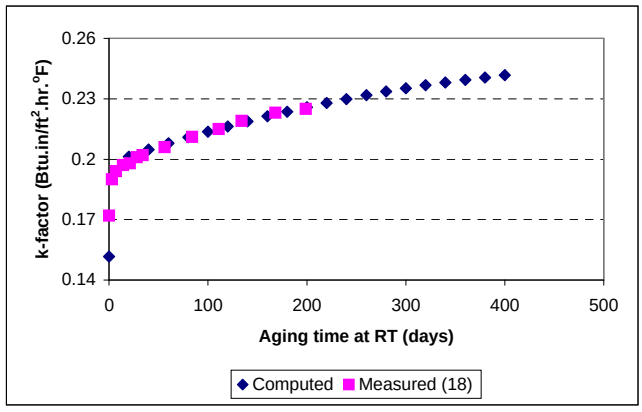



Figure 3. Computed and measured k-factor of skin layer

Figures 4 and 5 show the partial pressures of $N_2$, $O_2$, and HCFC-142b as a function of depth in a 2" (50 mm) thick foam after 3 and 14 days of aging of fresh boards calculated using the parameters listed in Table 1 for XPS #1. Clearly air diffuses in rapidly, with the highest concentrations near the surface. This will decrease the R-value of the board and that of any slice cut from the surface layer, while leaving the core layer essentially untouched in the time-period shown. If one was to cut a surface slice from boards of age 3 to 14 days since manufacturing (the window available in the S770 method), it is clear that, $R_{Slice,\ initial}$ would be much lower than $R_{Product,\ initial}$ even if $R_{Slice,\ initial}$ is measured instantaneously after slicing.

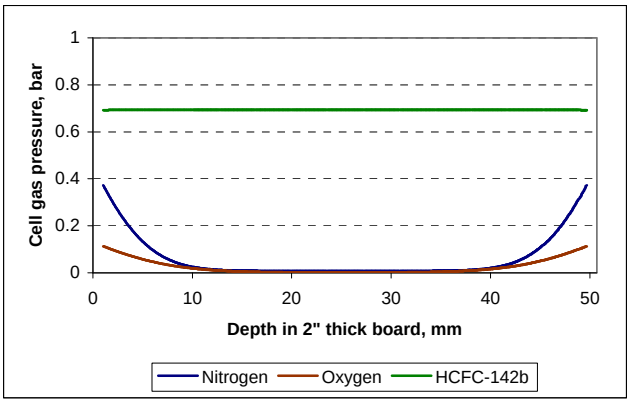



Figure 4. Cell gas pressure after 3 days in XPS #1

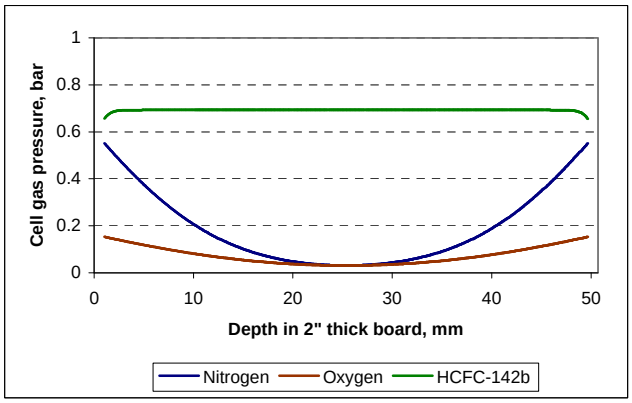



Figure 5. Cell gas pressure after 14 days in XPS #1

Figures 6 and 7 show the effect of the elapsed time between the start of slicing and the start of R-value measurement on the thin slice (i.e. start of $R_{Slice,\ initial}$ measurement) on % bias in the S770 predicted LTTR value for a 3-day-old and 14-day-old XPS board respectively. The % bias is calculated using the 5-year aged R-value from aging simulations on full thickness board as the true LTTR value. After all, as demonstrated by Figure 1, full thickness aging simulation matches experimentally measured R-value very well. Knowing that it takes 45 minutes to an hour to measure R-value of a board, we can see that even if one was extremely expedient in cutting all the surface slices and immediately putting them in a thermal conductivity (k-factor) measurement instrument to measure $R_{Slice,\ initial}$, one will have a positive bias of over 10% irrespective of the age (3-14 days) of the board with the S770 method. The % bias increases as time between start of slicing and start of R-value measurement on thin slice increases. The bias decreases as the thickness of the slice in increased, going to about 3% for 18 mm and about 1% for 24 mm slice in the window for measurement. Of course use of thicker slice decreases the attractiveness of the method as an aging acceleration method.

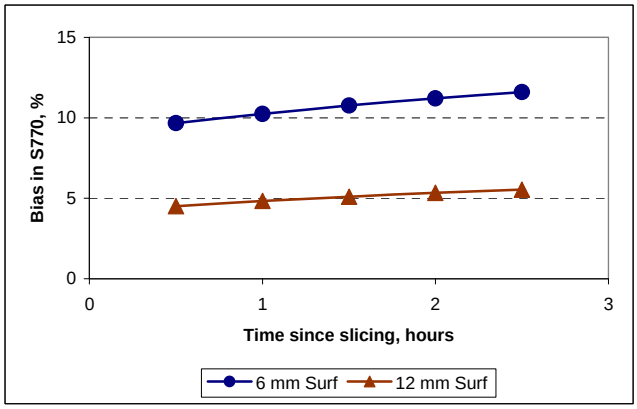



Figure 6. S770 bias when using 3-day-old XPS #1

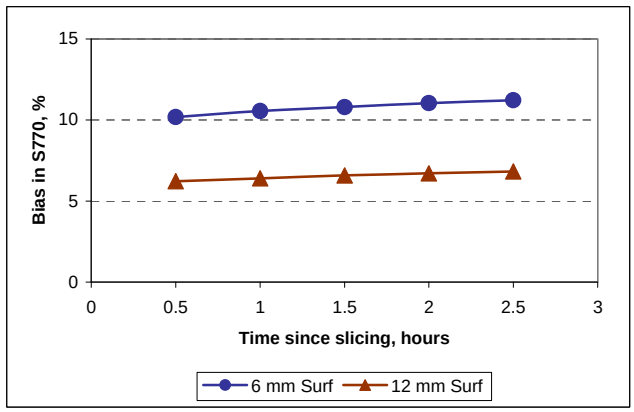



Figure 7. S770 bias when using 14-day-old XPS #1

Much of the high bias in Figures 6 & 7 is attributable to the low value of $R_{Slice,\ initial}$, in comparison to $R_{Product,\ initial}$ and not because of a high value for $R_{Slice,\ aged}$ (see Eq.2). $R_{Slice,\ initial}$, should be extremely close to $R_{Product,\ initial}$ for any slicing and

scaling based method to work. For XPS #1 board, it is significantly lower, by about the same factor as the bias, due to the phenomena shown in Figures 4 and 5, i.e., rapid ingress of air near the surface of the board. Actually this was understood by those developing the S770 method as section A4.5 of the S770 standard Appendix A4, titled "limitations for the use of the scaling equation," reads "*once cut, the thin layers age very rapidly. The aging factor derived by comparing thermal resistivity of the layers at the calculated testing points to their initial resistivity, can only be applied to the original full board initial thermal resistance if the initial measurements of the board and slices correspond to the same scaled age, measured from the time of cutting*." However, the current method does not require this. If this was required in the method, it would make $R_{Slice,\ initial}$, to be the same as $R_{Product,\ initial}$ which would mean LTTR = $R_{Slice,\ aged,}$ and this is exactly what C1303 does.

There is some discussion that perhaps the bias in S770 can be reduced if the method required the use of an average of the surface and core aging factor or the lower of the surface and core aging factor. Figure 8 shows the % bias when a 6 mm core slice, 6 mm surface slice, 12 mm core slice and 12 mm surface slice are used for a 3-day-old board. Figure 9 shows the same thing for a 14-day-old XPS board. Though the bias is low for the 6 mm core slice, the exact bias is a strong function of elapsed time between start of slicing and start of R-value measurement on the thin slice. Use of an average aging factor will still give a high bias for a 6 mm slice and near zero bias for 12 mm slice measured on the 14-day-old board. As demonstrated with calculations on boards XPS #2 and XPS #3, the exact slice thickness and age of the board where use of an average aging factor will yield near zero bias will change depending on the characteristics of the board.

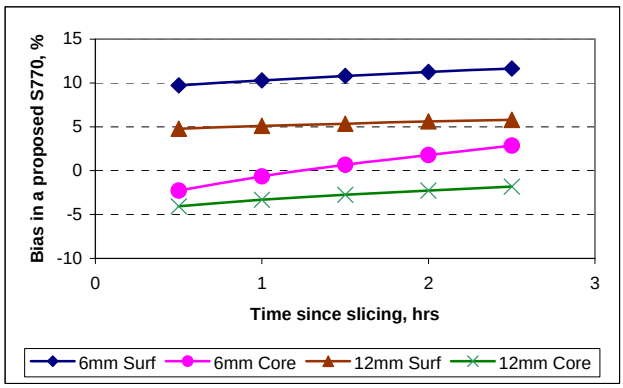



Fig 8. Modified S770 bias when using 3-day-old XPS #1

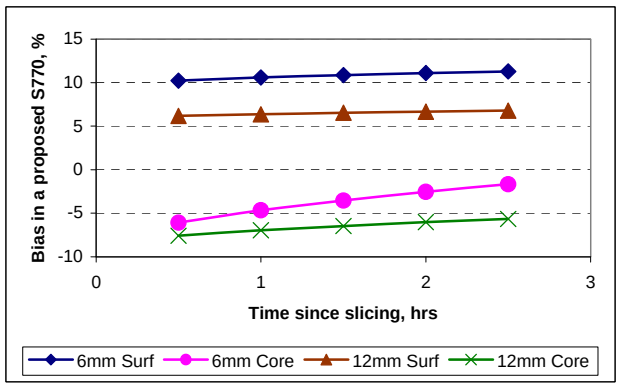



Fig 9. Modified S770 bias using 14-day-old XPS #1

The bias using the C1303 test method for XPS#1 board is shown in Figure 10. There was no significant difference between samples that were 3 and 14 days old. This suggests that the use of core slices will give a negative bias, i.e., under-prediction of the LTTR value for this XPS board which has a diffusion retarding skin. Use of surface slice on the other hand gives very low bias. The aging equivalence calculated using Eq. 4 for 9 and 12 mm slices is 104% for the 14-day-old board and 101% for the 3-day-old which is well within the 90-110% range currently stipulated in the method (10). Rapid diffusion of air causes a significant aging of the surface slice of the 14-day-old board as compared to the 3-day-old board leading to a larger difference between $(k_1 / k_2)_{Core}$ and $(k_1 / k_2)_{Surface}$ for the older board. This results in a larger aging equivalence for the older board.

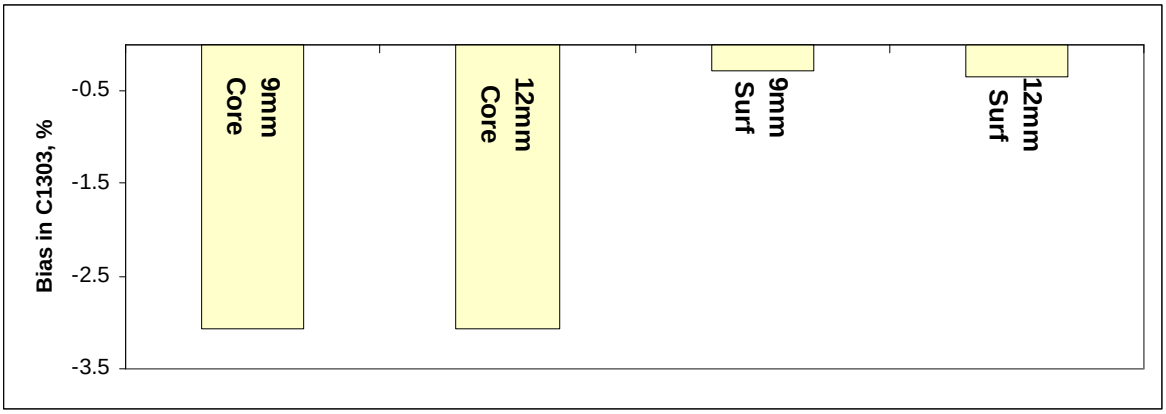



Figure 10. C1303 bias for different slice thickness and slice location

Results on another board XPS#2, show exactly the same behavior as XPS #1 except the S770 bias is even larger. This can be the seen by comparing Figure 11 with Figure 7. The reason behind the higher bias for XPS #2 can be seen by comparing Fig. 12 with Fig 5, which shows the cell gas pressure as a function of foam depth. With no diffusion retarding skin layer and faster $D_{eff}$ for $N_2$, $O_2$ and HCFC-142b, there is even a higher disparity between $R_{Slice,\ initial}$ and $R_{Product,\ initial}$. This leads to a higher bias as compared to the XPS #1. As can be seen from Figure 13, a S770 LTTR calculated in using average or the lower of the surface and core slices will not consistently (compare to Figures 8 & 9) lead to near zero bias. As expected, with no skin layer, this board is homogeneous and C1303 gives no bias at all.

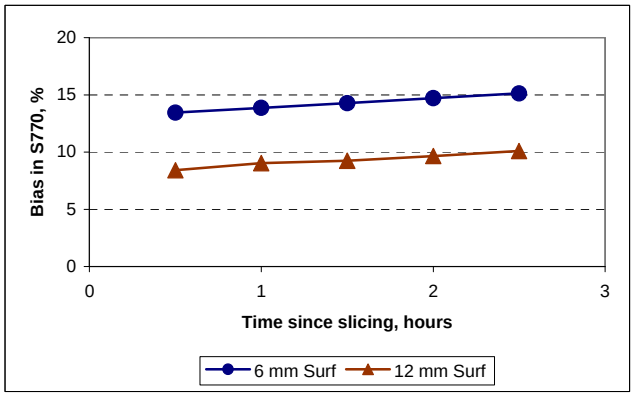



Fig. 11. S770 bias using 14-day-old XPS #2

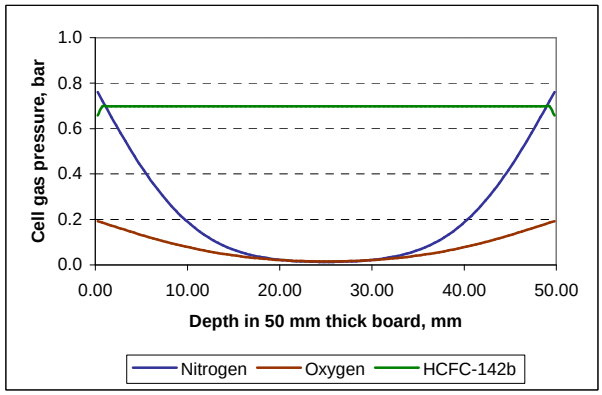



Fig. 12. Cell gas pressure after 14 days in XPS #2

Board XPS #3 is different than boards #1 and #2 as it contains a relatively fast diffusing blowing agent, HCFC-22, along with the slower diffusing HCFC-142b. As seen in Figure 14, S770 bias is the largest for XPS #3, even though it has a diffusion retarding skin layer. The reasons behind this can be extracted from Figure 15 which shows that in addition to air coming in rapidly, HCFC-22 is leaving the board rapidly. This combination leads to a high value for $R_{Product,\ initial}$ / $R_{Slice,\ initial}$ for all conditions within the S770 window. Once again Figure 16 suggests that simple changes such as average or lower of core and surface slices are not very persuasive. The bias in C1303 method was low, about -1% for core slices and nearly zero for surface slices. Age of the board did not have any effect on C1303 bias. The calculated aging equivalence though is significantly affected by age of the board. It was 103% for 3-day-old board whereas it was 107.5% for 14-days-old board. As discussed earlier, this dependency of aging equivalence on the age of the board is attributable to the much higher level of aging of the surface layer of 14-day-old board as compared to 3-day-old board. All the illustrations for XPS #2 and #3 are shown for only the 14-day-old board, the trends shown earlier for XPS #1 for 3-day and 14-day-old samples apply here too.

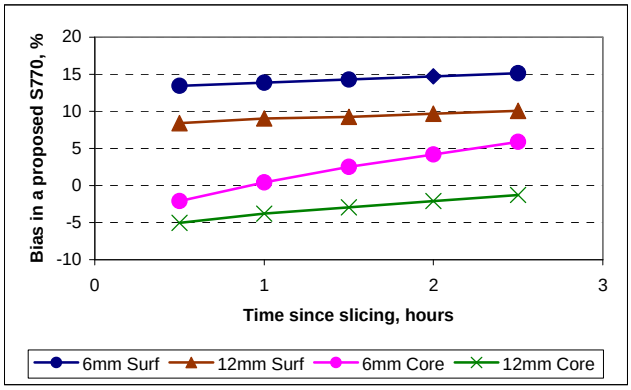



Fig 13. Bias in a proposed S770 for 14-day-old XPS #2

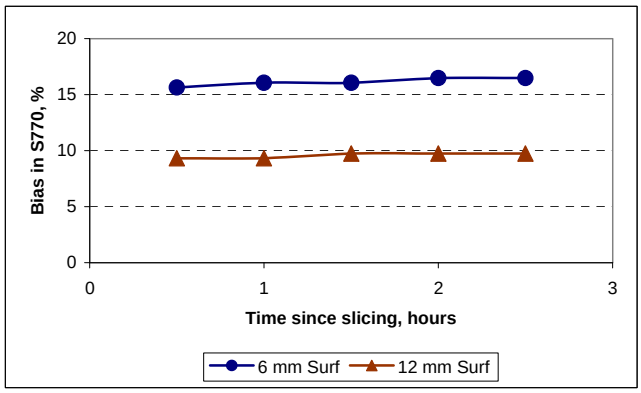



Fig 14. S770 bias when using 14-day-old XPS #3

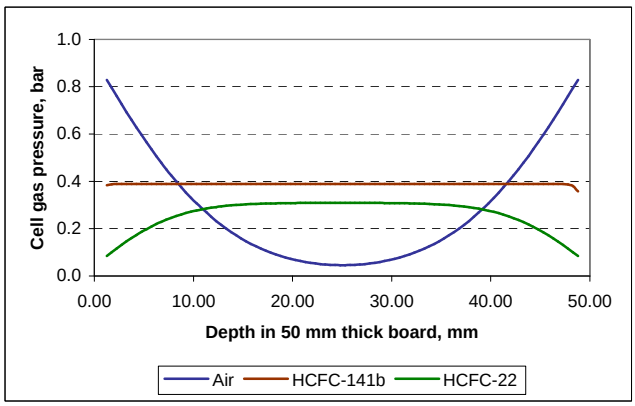



Fig 15. Cell gas pressure in XPS #3

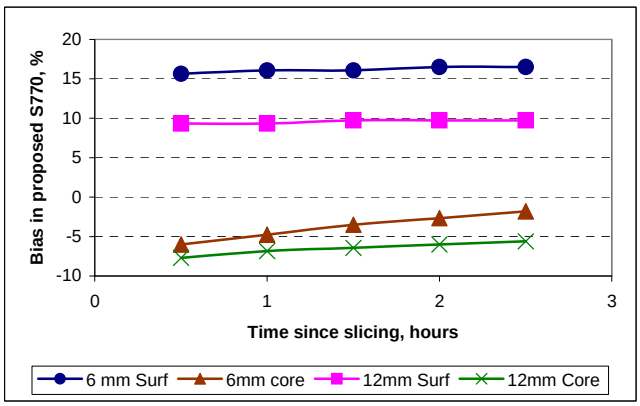



Fig 16. Bias in a proposed S770 for 14-day-old XPS #3

Even though we have used only a couple sets of data to illustrate the reasons behind high bias for XPS board with the current S770 method, the trends are irrefutable. This is because the underlying cause, relatively fast diffusion of air through XPS board is irrefutable. It is very unlikely that short of increasing the slice thickness to something like 25 mm or higher, simple practical changes to the current S770, such as tightening the time windows, taking average of core and surface aging factor will lead to a consistent near zero bias for XPS boards. C1303 on the other hand appears to predict the LTTR with little bias, even for quite heterogeneous boards, as long as the higher of the core, surface or mixed slice value ($R_{Slice,\ aged}$) is used.

## CALCULATIONS ON POLYISO BOARDS

Diffusion coefficients and other parameters chosen for the illustration of polyiso board behavior are shown in Table 2. polyiso #1 is board #2 from Huntsman's 2002 API paper (2). As seen in Figure 1, there is over 5 years of room temperature aging data on it. As explained below, polyiso #2 is created here to explain prior studies of the S770 method with polyiso boards. Polyiso #3 is board #1 from our 2002 paper (2).

**Table 2: Input parameters used to model various Polyiso foam boards**

| Gas | Effective Diffusion Coefficient, $D_{eff}$ ($10^{-12}$ $m^2s^{-1}$) at RT | | |
|---|---|---|---|
| | **Polyiso # 1** | **Polyiso # 2** | **Polyiso #3** |
| Air | 2.0 | 3.77 | 3.77 |
| $CO_2$ | 124 | 118 | 118 |
| Iso-pentane | 0.018 | 0.0522 | 0.0522 |
| Cyclo-pentane | 0.043 | 0.128 | 0.128 |
| | | | |

| Other parameters | | | |
|---|---|---|---|
| Density, pcf (Kg/m$^3$) | 1.8 (29) | 1.8 (29) | 1.7 (27) |
| Thickness, inch (mm) | 1.6 (40) | 1.6 (40) | 2 (50) |
| Skin Factor | 1/12 | 1 | 1/23 |

Figure 17 shows the partial pressures of air, $CO_2$, cyclopentane and isopentane as a function of depth in the 1.5" (40 mm) thick board after to 14 days of aging. They were calculated using the parameters listed in Table 2 for polyiso #1. Clearly air diffuses slowly into this polyiso board, whereas, $CO_2$ diffuses out more quickly. Figure 18 shows the effect of slice age on the % bias in S770 results using 6 and 12 mm slices. There is a small negative S770 bias for this polyiso board. This is because of the relatively slower $D_{eff}$ for air through polyiso board and also because of the presence of the skin layer retards diffusion to a point that $R_{Slice,\ initial}$ is very close to $R_{Product,\ initial}$ and thus S770 LTTR value is closer to the computed LTTR value on full thickness product. There is no significant difference in S770 results between 3-day-old and 14-day-old board.

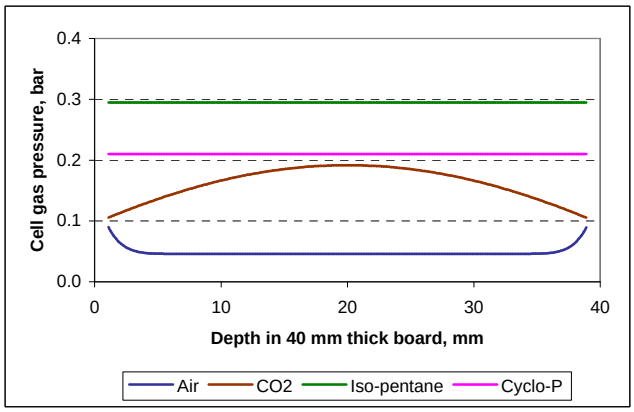



Fig 17. Cell gas pressure in the 14-day-old polyiso #1

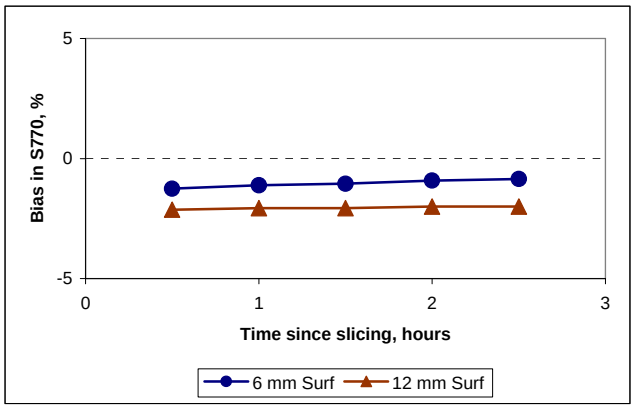



Fig 18. S770 bias when using a 14-day-old polyiso #1

The bias using the C1303 test method for this polyiso board #1 is shown in Figure 19. Core slices yield a significant (5.6%) negative bias, i.e., under-prediction of the LTTR value for this polyiso board which has a diffusion retarding skin. Use of a surface slice on the other hand gives relatively smaller, though still negative bias. The aging equivalence calculated using Eq. 4 for 9 and 12 mm slices are 104.8% and 104.4% respectively which is well within the 90-110 range currently stipulated in the method (10). Age of the board between 3 and 14 days did not have any effect on the C1303 bias or aging equivalence.

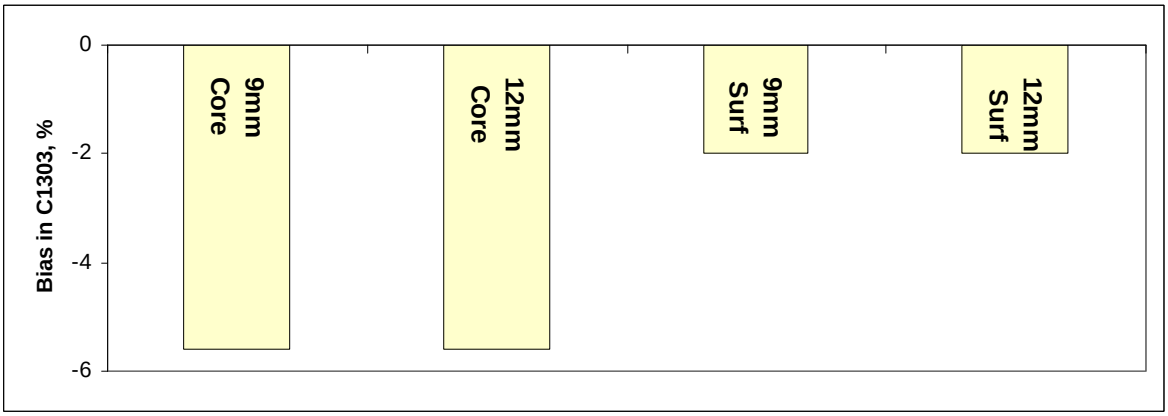



Figure 19. C1303 bias for different slice thickness and slice location

This raises the question why the ASTM C1289 precision and bias study in Reference 8 and the data reported in Reference 9 gave a positive bias for polyiso board. There could be several ways that a polyiso board can yield a positive bias. Let's look at what happens if the skin factor of the polyiso #1 board is made to be one, i.e., no skin on the board. Figure 20 shows the cell gas pressure after 14 days from manufacturing for this hypothetical board, dubbed polyiso #2. Indeed a lot more air diffuses into the board and raises both $R_{Product,\ initial}$ and $R_{Slice,\ initial}$ which results in a positive bias for the S770 results (see Figure 21). The level of bias shown in Fig 21 is similar to those measured in Reference 8-9. Of course, C1303 will yield zero bias as this is a homogeneous board.

There is possibly another explanation to the small positive bias seen for some polyiso boards. Polyiso board production is an exothermic process and the boards are typically bundled into 4' x 8' x 4' boards soon after production. This can keep temperature elevated for hours, especially away from edges of the stack. Fig. 22 shows the cell gas analysis of polyiso #2 board after it is exposed to a temperature of 100°C for 12 hours. The cell gas distribution in the foam looks very similar to that seen in Fig 20. We must remember that polyiso #2 board is a hypothetical board with diffusion coefficients and initial cell gas pressure being exactly the same as polyiso #1 but the skin factor forced to be one, meaning no diffusion retardation from skin. If the board polyiso #1 was aged for 12 hours at 100°C, the cell gas composition in the board will be significantly different, as seen in Figure 23. For a board with an initial cell gas composition shown in Figure 23 and diffusion characteristics of polyiso #1, the calculated bias is less than 1% for all the conditions encompassed in the S770 test.

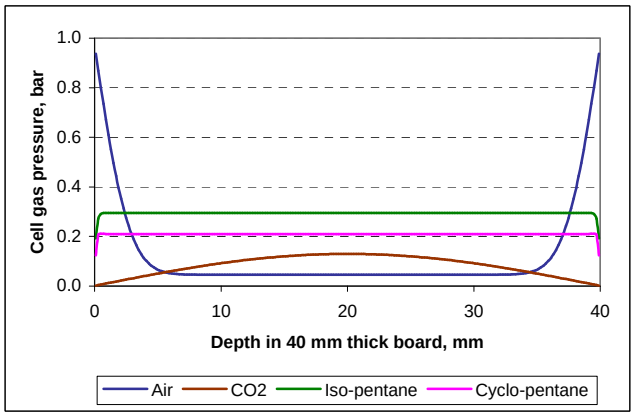
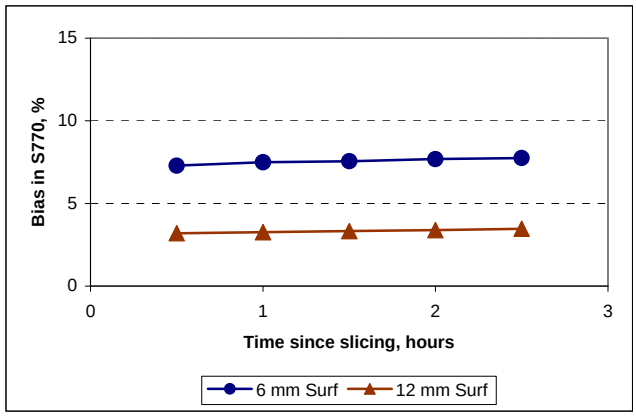



Fig 20. Cell gas pressure in the 14-day-old polyiso #2




Figures 21. S770 bias when using a 14-day-old polyiso #2

Figure 24 shows the S770 bias for the board polyiso #3 which is the same as the board #1 from Reference 2. This board was made on a commercial laminator in the USA and thus had seen the heat history discussed above. LTTR number of this board was tested in 2002 following the S770 method (2). As shown in Reference 2, the measured LTTR value for this board using a 9 mm slice thickness was 5.69 and the calculated five year full thickness aged R-value was 5.65 which gave it a bias of +0.7%. Figure 24 suggests a similar bias for a 9mm slice. This further validates the computational capability of Agesim.

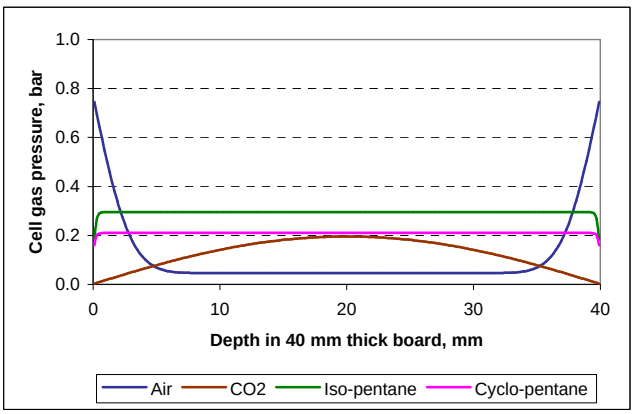



Fig. 22. Cell gas pressure in Polyiso #2

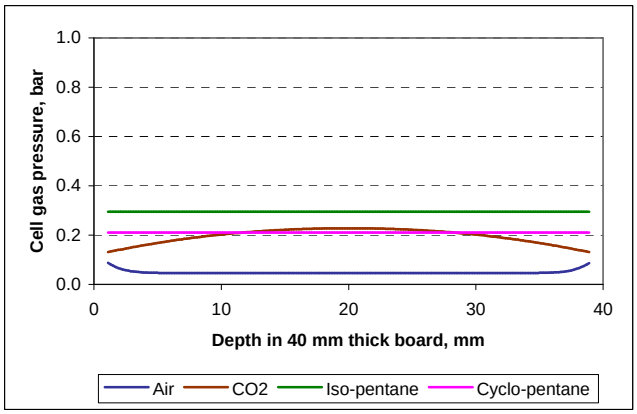



Figure 23. Cell gas pressure in stack cured polyiso #1

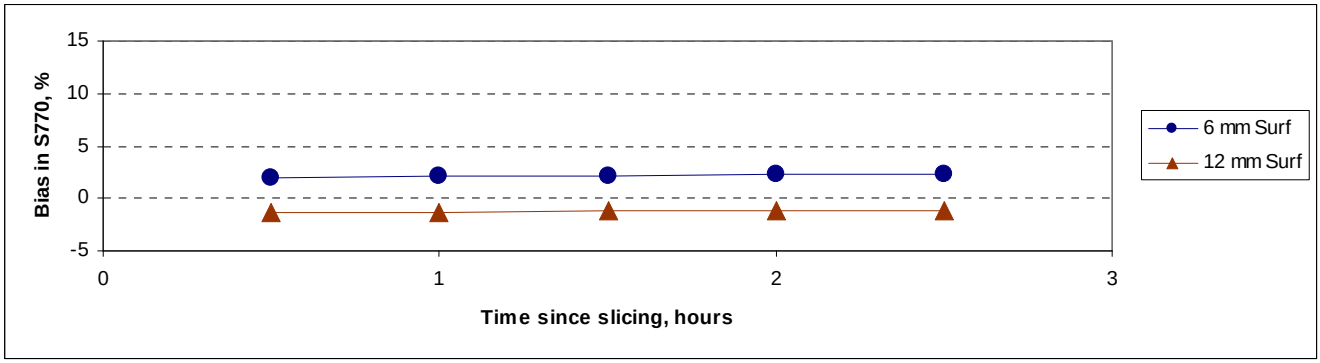



Figure 24. S770 Bias for polyiso #3

The bias using the C1303 test method for the polyiso #3 board computes at -6.3 % using core slices and -3.5% with surface slices for both 9 and 12 mm slices. This is a relatively large under-prediction despite the fact that the aging equivalence calculated using Eq. 4 for 9 and 12 mm slice is about 106.5% which is still within the 90-110% limits currently stipulated in the method (10). Once again, age of the board between 3 and 14 days did not have any effect on C1303 bias or aging equivalence.

In addition to the absence of a diffusion retarding skin layer, some of the other reasons behind any measured positive bias for polyiso boards could be excessive vibration during slicing and the errors introduced because of inclusion of a facer on the surface slice. Excessive vibration during slicing destroys the integrity of the cells next to the surface being cut. This will reduce both $R_{Slice,\ initial}$ and $R_{slice,aged}$ but generally the ratio $R_{slice,\ aged}$ to $R_{slice\text{-}initial}$ will be larger for a poorly cut sample compared to a properly cut sample leading to a positive bias. In addition, polyiso boards always have a facer on the surface which is a non-aging, non-foam component. Unlike the C1303 method, S770 does not exclude the facer in the slice thickness and aging time calculations. Depending on the thickness of the facer as compared to the slice thickness, this can contribute to a 1-2 % positive bias for typical facers.

The above discussions suggest that the S770 method should give a low bias, ±2%, for polyiso board with a diffusion retarding skin. However, there are situations which could lead to a larger positive bias even for polyiso boards. We need to remember that due to the temperature gradient across the thickness of the polyiso board during manufacturing, the density next to the facer is generally higher and the cell size lower than in the center of the foam (12). This should lead to a diffusion retarding skin layer in nominal polyiso production. With the ability to control the degree of overpack, facer/laminator temperature, the residence time in the laminator, and also numerous formulation variables such as level or type of surfactants, catalysts, and blowing agents, a polyiso manufacturer has the ability to make boards with good surface barrier properties. It is essential that the LTTR method used by the industry be such that the polyiso manufacturers have incentives to produce such beneficial products. The above calculations suggest that the C1303 method can yield a high negative bias for such products even when the highest of the LTTR values calculated from the surface, core or a mixed slice ($R_{Slice,\ aged}$) is used.

## LIMILTATIONS OF THE AGESIM

It is important to keep in mind that like any mathematical model, the thermal aging simulation software, Agesim, makes many assumptions. Fickian diffusion through a composite layer consisting of homogeneous core and a homogeneous skin layer; concentration independent diffusion coefficients; homogeneous solid conduction and radiative heat transfer components of thermal conductivity; and no thermally destroyed surface layer in any slices are the most noteworthy assumptions for Agesim. Though slight deviations from these assumptions will keep the predicted trends intact, it is likely that larger deviations, especially those having cumulative impact in one direction would make the prediction questionable.

At the same time, much of what is discussed in this paper can be explained simply by thinking through the consequences of diffusion of a relatively fast diffusing gas like air into the foam boards from the outside in and the relatively slow diffusion of most widely used blowing agents. The reason behind a relatively high positive bias for XPS boards with the S770 method is that its $D_{eff}$ for air is such that it interacts with the current S770 measurement window in a way that we get large, varying positive bias. Typically, such is not the case with polyiso foam.

## CONCLUSIONS

1. The current S770 method does not appear to be an appropriate method to measure LTTR for XPS boards. Except for raising the slice thickness to 25 mm or more, there does not appear to be any practical modification to the current S770 method to make it amenable to XPS board.

2. The current S770 method is a reasonable method for measuring the LTTR value of polyiso boards. It appears that high temperature during storage of the board soon after manufacturing, poor skin layer and inclusion of the facer layer in the surface slice could be responsible in some combination for much of the relatively smaller (as compared to XPS) positive bias. Simple modifications to the S770 method, namely raising the slice thickness to 9 mm of foam, to reduce the impact of the thermally destroyed surface layer and non-aging facer layer will reduce the bias significantly for any polyiso board.

3. The C1303 method will under-predict the LTTR value of any foam containing a diffusion retarding skin. Approaches to reduce this under-prediction need to be considered by the ASTM C 1303 task group.



## ACKNOWLEDGEMENTS

The authors acknowledge the work of Dr. Guy Biesmans, Dr. Kristof Dedecker and numerous others who contributed to development of the thermal aging simulation software, Agesim. The authors would also like to thank all the associates at Huntsman Advanced Technology Center who helped set up the laboratories and testing facilities in time to make this type of work possible.

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## BIOGRAPHIES

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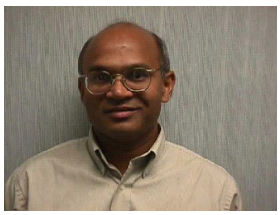

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