

# Long Term Thermal Resistance of Pentane Blown Polyisocyanurate Laminate Boards

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## ABSTRACT

The accurate estimation of the thermal performance of insulation products over their expected lifetime has been a recognized challenge for over twenty years. This is because the lifetime of such products is long (10-25 years), thermal aging is caused by the diffusion of a multitude of gases, and the insulation product is not homogeneous. Two classical approaches to accelerating diffusion controlled phenomena, namely aging at higher temperature and aging a thin slice have limitations when applied to insulation materials like polyisocyanurate (PIR) laminate board. Raising temperature does not change the diffusion coefficients of all the different gases involved in the aging process at the same rate. In the case of pentane blown PIR board, these gases are CO<sub>2</sub>, air and the different pentane isomers used to blow the foam. Thin slicing of core foam and scaling techniques, such as those in the ASTM C 1303 -00 procedure, do not account for the fact that PIR laminate boards are denser at the surface layer than in the core foam.

A multitude of mathematical modeling and calculation algorithms, such as the effective diffusion model and the distributed parameter continuum (DIPAC) model, have been developed in an attempt to overcome the above stated limitations. However, these models become too complex and onerous for any routine, industry-wide adaptation.

Polyisocyanurate insulation manufacturers in the USA and Canada have wrestled with the above issues for many years and have now identified a test method, CAN/ULC-S770-00, which appears to balance the need for accuracy and simplicity. This method defines the long-term thermal resistance (LTTR) as the value measured after 5-year storage in a laboratory environment. The procedure used to accelerate diffusion is based on a thin slicing and scaling of the core foam and the surface layers.

One of the objectives of this paper is to compare the LTTR value predicted by the CAN/ULC-S770-00 Test Method to those obtained from the mathematical modeling and calculation algorithms developed by Huntsman Polyurethanes. Another is to compare the thermal resistance of pentane-blown PIR laminate boards aged in a laboratory environment since early 1998 to those obtained from the mathematical modeling and calculation algorithms developed by Huntsman Polyurethanes. Through such comparisons, it is shown that the CAN/ULC-S770-00 Method, which takes only weeks to complete, indeed predicts the thermal performance of a laminate board after almost five years of actual aging in a laboratory environment.

In short, this paper provides credibility to the new LTTR Method identified by the boardstock industry and thus helps maintain PIR boards as the insulation material of choice for the construction industry.

## INTRODUCTION

Polyisocyanurate laminate boards are closed-cell foam insulation products used extensively in commercial, industrial, and residential applications to save energy. They are filled with insulating gases and thus have the highest thermal resistance of any conventional insulation, excluding those containing vacuum. Over time, the thermal resistance of the foam laminate changes as air components diffuse into the foam and the insulating gases diffuse out. Since air has a poorer thermal resistance than the blowing agents, thermal aging occurs. However, the diffusion for some gas components is very slow, therefore the total aging process can take many decades [1] and stops only when equilibrium concentrations are reached for each gas.

The long duration of the aging process makes the estimation of thermal performance over the expected life of insulation products a challenge. This is especially true for products used in roofing applications because such products are exposed to a myriad of different field conditions, from very cold to very hot/humid temperatures over a long life span, usually decades. Long-term roof field test data from different locations, with different climates and seasons is surprisingly very limited [2,3].

For practical purposes, laboratory monitoring is used to evaluate the long term performance of roofing insulation. Despite much effort over the past 30 years, there is still some disagreement about what process should be used to accelerate the aging process [3, 4]. The two classical approaches to accelerate diffusion-controlled phenomena are either through elevated temperature or by reducing the thickness of tested foam layers. When applied to determining the LTTR of PIR laminate boards used in roofing applications, each of the two approaches offer some pluses and minuses. Although the elevated temperature approach is much favored in many European countries [5], some researchers object to this approach as it fails to consider that the diffusion characteristics of the different gases do not change at the same rate as the temperature is changed [3,6].

On the other hand, thin slicing of core foam and scaling techniques such as those in the ASTM C 1303-00 procedure do not account for the fact that many PIR boards are denser at the surface layer than in the core foam [4,7].

In order to overcome the above stated limitations of the two classical approaches, a multitude of mathematical modeling and calculation algorithms have been developed over the last 30 years [1]. Examples of such models are the effective diffusion model, the membrane permeation model and the distributed parameter continuum (DIPAC) model [4,7]. Although many of these models and calculation algorithms have been demonstrated to be accurate and capable, in general, they require an extensive range of input parameters. For example, the diffusion model developed by Huntsman Polyurethanes to predict the aging behavior of laminate board requires as input parameters the effective diffusion coefficients of all the blowing agents through the foam and facers, the initial cell gas composition, and the thermodynamic properties of the pure blowing agents and their mixtures.

The determination of such parameters is expensive and time-consuming, especially since it depends on the foam density, the cell morphology etc. [9, 10]. Cell orientation and density will vary within a laminate, among different manufacturers and between plant sites, making it necessary to measure such parameters for each individual foam sample. Clearly, this modeling methodology is too

complex and onerous to be used on a routine basis by industrial laboratories or standard setting bodies.

Clearly then the development of an efficient, realistic laboratory test method to assess the in-service long-term thermal resistance (LTTR) is needed. This will help building specifiers make informed design recommendations and purchasing decisions, and provide for fair comparisons with other types of insulations. Also, such a procedure would greatly reduce the time and cost of monitoring the performance of existing products and speed up the assessment of formulation/processing changes.

## CAN/ULC S770-00

After about 10 years of research, a prescriptive method for determining the long term R-value using the slicing and scaling method, CAN/ULC-S770-00 (hence forth referred as S770) has been recently approved for use in Canada and proposed for use in USA [11, 12]. In developing the S770 test, the Canadian Plastics Industry Association decided that LTTR shall be defined as the time-weighted average of thermal resistance over 15 years at a given thickness. This is because the average useful life of a PIR roofing insulation is around 15 years. Based on some approximations, the method states that the 15 years time-weighted average is equivalent to the actual R-value after 5 years [13]. Thus the LTTR of a thermal insulating foam product is defined in the S-770 standard as the thermal resistance measured under standard laboratory conditions ( $23 \pm 2^\circ\text{C}$  and  $50 \pm 10\% \text{ RH}$ ) - equivalent to the resistance after a 5-year storage period in a room.

The procedure used to accelerate diffusion is based on thin slicing and scaling of the core foam and surface layers. This technique is based on the fact that the gas diffusion occurs at a rate inversely proportional to the square of the thickness of the product. To determine how long a thin slice of foam should be aged to correlate to the actual 5-year aging of a PIR board, the following equation is used:

$$\frac{t_1}{t_2} = \left( \frac{L_1}{L_2} \right)^2 \quad (1)$$

where  $t_1$  is the time in days that the thin slice should be aged,  $t_2 = 1825$  days (= 5 years),  $L_1$  is the thickness of the thin slice (6 - 10 mm), and  $L_2$  is the thickness of the polyiso board in mm. Thus to measure LTTR of a 2" laminate board using a 6.5 mm thin slice, the total time required is 25.5 days and significantly shorter than 5 years.

## OBJECTIVE

One objective of this paper is to compare the LTTR value predicted by the CAN/ULC-S770-00 test method to

one obtained from the mathematical modeling and calculation algorithms developed by Huntsman Polyurethanes. Another is to compare the thermal resistance of pentane blown PIR laminate boards aged in an uncontrolled ambient laboratory environment since early 1998 to those obtained from the mathematical modeling and calculation algorithms developed by Huntsman Polyurethanes.

## EXPERIMENTAL

### Materials

Two Polyiso foam rigid insulation boards, each blown with cyclopentane, isopentane and water, were used in this study. Board #1 is a 2" (5.1 cm) thick experimental board made at a U. S. laminator. Board #2 is a 1.5" (3.8 cm) thick experimental board made at the Huntsman Polyurethanes' small-scale laminator in Ternate, Italy in February 1998. The formulation and processing conditions used to make Board #2 are described generically in another paper [14]. Although the formulations for the two boards are somewhat similar, the processing conditions were not identical. A typical black fiberglass facer, used to make roofing products, was used for both boards.

### Thermal Resistance Measurements and Foam Aging

The foam thermal resistance was measured according to ASTM C518 with a mean plate temperature of 75°F and temperature difference of 40°F. Foam aging was performed on 12" x 12" x full-thickness laminate or a 12" x 12" x 1" core foam.

### CAN/ULC S770 – 00 Methods

The following procedure was used to measure LTTR for the Board #1:

- Initial R-values were measured for the polyiso boards seven days after production.
- Immediately after R-value measurement, four thin surface slices and four thin core slices, each of about 6.5 mm thickness were cut from two of the samples.
- The surface slices were stacked together and initial R-value measured. The same was done for the core foam slices.
- The thin surface and core slices were stored individually in a temperature-controlled laboratory environment at 23°C ± 5°C, with the major surfaces of all the slices exposed to the ambient air.
- At the time intervals determined using equation (1), the surface and core foam slices were stacked separately and measured for R-value.
- An aging factor was calculated for the surface slices and the core foam slices. As the acceptance criteria defined in the method were met, the larger of the two

aging factors was multiplied by the initial R-value of the original polyiso samples to obtain an aged LTTR at that particular thickness.

Board #2 was made in February 1998, much before the S770 Method was accepted or even developed. As S770 must be done between 7-14 days after manufacture, we were unable to measure LTTR using S770 for Board #2.

### Cell Gas Analysis: Gas Chromatography

The cell gas content was analyzed quantitatively using a modified gas chromatograph [8]. Small foam cylinders (9 mm diameter, 2-3 cm length) were drilled out of the foam samples using a cork bore. These cylinders were subsequently crushed and ground by a special device which collects escaping gases into an injection system. The device was preheated at 100°C to allow for vaporization of all the blowing agents present in the foam cells. Any blowing agent absorbed in the foam matrix was not released during this short crushing period. Subsequent injection on a gas chromatograph allowed for quantitative and qualitative analysis of all gases. Calibration was done via measuring the retention time and response area of a well-defined amount for every relevant gas. Based on the weight of the original foam cylinder, the foam density and closed cell content and the gas composition was expressed as a weight percent or as a cell gas pressure.

### Thermal Aging Simulations: Agesim

As discussed earlier, the change in cell gas composition over time and related change in gas thermal conductivity is quite a complex phenomenon. Different gases have different effective diffusion rates ( $\text{CO}_2 > \text{air} > \text{BA}$ ), which will also depend on foam morphology and temperature. Moreover, blowing agents can undergo condensation and interact with the foam matrix. The exact calculation of cell gas composition and gas thermal conductivity (k-gas), as a function of time and position in the foam, is done within the thermal aging software developed by Huntsman Polyurethanes called Agesim. A basic description of the calculations is given below.

The first step in the calculation is to define an initial cell gas composition for a given foam sample. This can be calculated on the basis of the formulation or, even better, be measured on the basis of the above described cell gas analysis technique. Initial gas thermal conductivity is calculated on the basis of the Wassiljewa equation (Lindsay-Bromley calculation of gas interaction parameter). The foam sample is subsequently divided into small finite elements, which for all the gases have a well-defined temperature and diffusion coefficient. The experimental determination of these diffusion coefficients is described later. If the foam sample is subjected to a

temperature gradient, diffusion coefficients vary in each finite element, depending on the temperature within each element. If the facers act as diffusion barriers, the exact diffusion coefficient of each gas through the facer can also be specified. Condensation is also assessed for each finite element, again depending on its temperature. In each time step, every element will undergo diffusion. After each time step, the gas-liquid equilibrium is first reassessed and k-gas is subsequently calculated. This procedure (diffusion – condensation – k-gas) is then repeated until the complete time period of interest is covered. The main output consists of graphs of (position-averaged) cell gas pressure and k-gas as a function of time, and as a function of position at a given time.

### Effective Diffusion Coefficient Determination

Effective diffusion coefficients ( $D_{eff}$ ) and their temperature dependence are a very critical input parameter for obtaining a successful thermal resistance aging simulation. Two basic methods, both requiring cell gas analysis, were used at Huntsman to determine diffusion coefficients. In the first method, a relatively thin lambda block (e.g. 12 x 12 x 1 inch) was aged at a well-defined temperature. By performing cell gas analysis (before the gas has reached its equilibrium value), the  $D_{eff}$  of air and  $CO_2$  were determined via a back-calculation with Agesim.

This method is not suitable for blowing agents that diffuse very slowly. Instead, small cylinders were cut, aged for a given time at a certain temperature, and again subject to cell gas analysis. By taking into account the specific geometry of the cylinders, the  $D_{eff}$  of each blowing agent was calculated. The  $D_{eff}$  at different temperatures is described by an Arrhenius type of equation. Therefore, after measuring  $D_{eff}$  at two temperatures, 23°C and 70°C, the  $D_{eff}$  of the blowing agents over a wider temperature range was calculated [8].

In order to simulate the aging behavior of products like PIR roofing boards, where densified skin layer and/or surface density gradient have different diffusion characteristics than core foam, the role of the surface layer needs to be factored. This was done in the Agesim model by assuming a 1mm thick material with  $D_{eff}$  different than that of core foam. The  $D_{eff}$  of this 1 mm material was expressed as a fraction of the  $D_{eff}$  of the core foam, i.e.,

$$D_{eff} \text{ through skin/facer} = X * D_{eff} \text{ through core foam} \quad (2)$$

Generally “X” is referred as the skin factor and was calculated iteratively by fitting measured k-factor aging curves of the laminate at elevated temperature (70°C) with those predicted by Agesim.

## RESULTS AND DISCUSSIONS

Table 1 shows the measured effective diffusion coefficient values obtained on the core foam taken from Board #1. These numbers are within the range of  $D_{eff}$  measured at Huntsman over the years.

Gas	Effective diffusion coefficients, $D_{eff}$ ( $10^{-12} \text{ m}^2 \text{ s}^{-1}$ )	
	73°F (23°C)	158°F (70°C)
$CO_2$	124	712
Air	3.77	71
Cyclopentane	0.128	0.418
Isopentane	0.052	0.159

Figure 1 compares the predicted thermal resistance at room temperature using the above  $D_{eff}$  obtained via Agesim to actual data from aging a 1” thick core foam taken from Board #1 at 23°C. As can be seen in Figure 1, the predicted and measured R-values show excellent agreement. The same was the case for core foam aged at 70°C. Given that  $D_{eff}$  were calculated using cell gas analysis alone, such agreement builds confidence in Agesim.

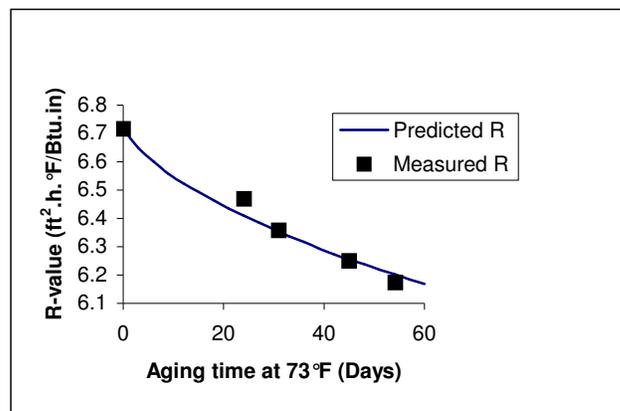


Figure 1: Predicted and measured thermal resistance for core foam aged at 73°F (23°C) - Board #1.

To obtain the skin factor “X”, a full laminate was aged at an elevated temperature of 158°F (70°C). Aging at 70°C was selected because at 23°C the full laminate aged very slowly, due to the skin/facer, and  $D_{eff}$  could not be determined within a reasonable period. The skin factor “X” was calculated for Board #1 by fitting the experimentally measured k-factor of laminate aged at 158°F (70°C) with that predicted by Agesim using the  $D_{eff}$  shown in Table 1. As seen in Figure 2, a skin factor of 23 gave a good fit between measured and predicted thermal resistance. The same was found to be the case for laminate

aged at 104°F (40°C) (Figure 3). These results suggest that aging of the 2" thick laminate Board #1 can be modeled as that of a 2" (50.6 mm) core foam with  $D_{eff}$  as shown in Table 1, and with two 1 mm thick skin layers on both sides with a  $D_{eff}$  of 1/23 of those shown in Table 1.

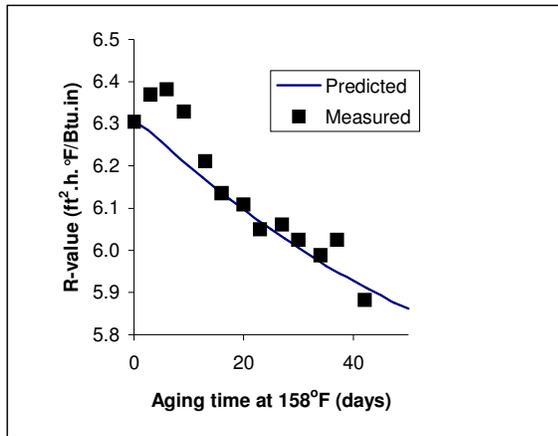


Figure 2: Predicted and measured thermal resistance for 2" thick laminate foam aged at 158°F (70°C) - Board #1.

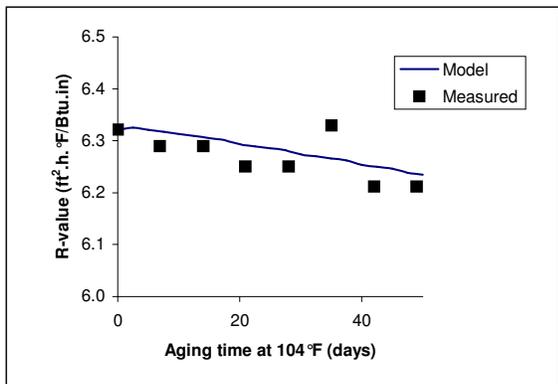


Figure 3: Predicted and measured thermal resistance for 2" thick laminate foam aged at 104°F (40°C) - Board #1.

The thermal resistance aging behavior of Board #1 at room temperature (23°C) for any period of time can now be calculated using Agesim. Figure 4 shows the thermal resistance behavior for Board #1 at 23°C over a 15-year period.

The 15-year time weighted average LTTR along with the 5-year instantaneous R-value were obtained from the aging curve shown in Figure 4. The LTTR value measured using S770 is also shown in Table 2. As discussed earlier, the S770 value is derived by multiplying the initial R-value with an aging factor. For Board #1, the initial R-value was measured as 6.4 and the aging factor was measured as 0.89 and 0.83 for surface and core layer respectively. As the aging factor calculated for surface

and core layers are less than 12% apart, the S770 LTTR for this board was calculated as  $6.4 \times 0.89 = 5.69$ .

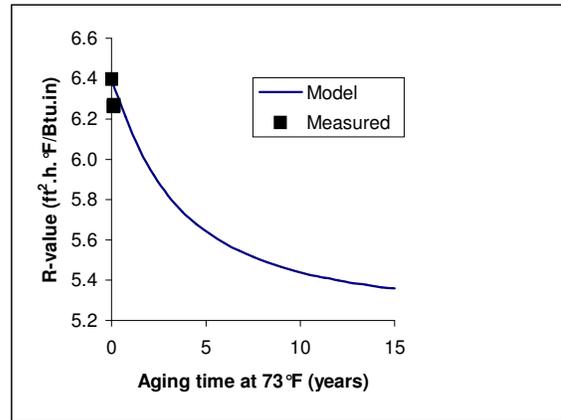


Figure 4: Predicted 15 year thermal resistance for 2" thick laminate foam aged at 73°F (23°C) - Board #1.

As seen in Table 2, the 3 LTTR values are within 1.5% of each other. This clearly suggests that for the Board #1 (a) the 15-year time-weighted average R-value was equivalent to the actual R-value after 5 years and (b) the LTTR measured by S770 is equivalent to those predicted Agesim.

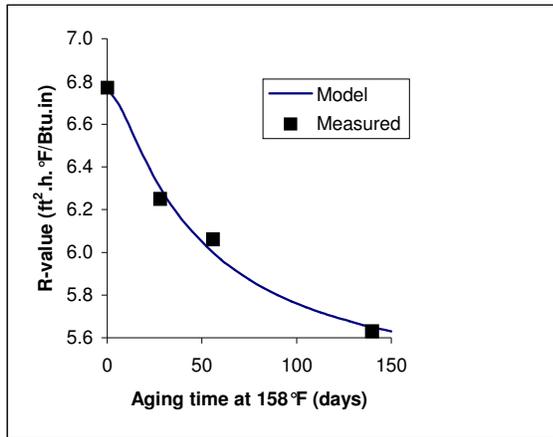
Method	R-value (ft <sup>2</sup> .hr.°F/Btu.in)
S770 measured	5.69
5 year predicted	5.65
15 year weighted average - predicted	5.61

Table 3 shows the measured effective diffusion coefficient values obtained on the core foam taken from Board #2. A comparison of core foam  $D_{eff}$  values for Boards #1 and #2 indicates that diffusion through Board #2 is almost half that through Board #1. This suggests that different processing conditions can indeed affect the thermal aging process significantly.

Gas	Effective diffusion coefficients, $D_{eff}$ ( $10^{-12} \text{ m}^2 \text{ s}^{-1}$ )	
	73°F (23°C)	158°F (70°C)
Air	2.02	40.3
Cyclopentane	0.043	0.137
Isopentane	0.018	0.073

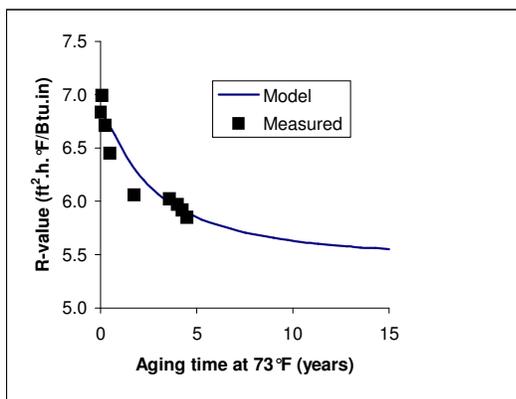
The skin factor "X" was calculated for Board #2 by fitting the experimentally measured k-factors for a laminate

aged at 158°F (70°C) with those predicted by Agesim using the  $D_{eff}$  shown in Table 2. As seen in Figure 5, a skin factor of 12 gave a good fit between measured and predicted thermal resistance. These results suggest that the aging of the 1.5" thick laminate Board #2 can be modeled as that of 1.5" thick core foam with  $D_{eff}$  as shown in Table 3 and a 1 mm thick skin layer on both sides with a  $D_{eff}$  of 1/12 of those shown in Table 3.



**Figure 5:** Predicted and measured thermal resistance for 1.5" thick laminate foam aged at 158°F (70°C) - Board #2.

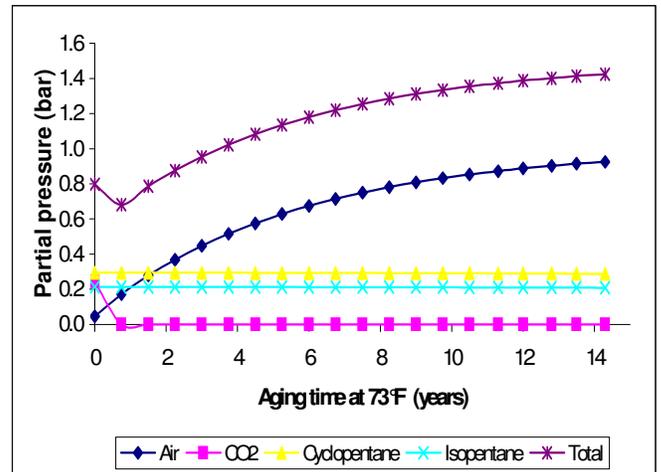
Based on the above input parameters, the thermal resistance aging of Board #2 over a 15-year period at room temperature was predicted and is shown in Figure 6. Figure 6 also shows the periodically measured R-value on the 4.5-year old laminate stored in unregulated ambient conditions. Figure 7 shows the corresponding changes in partial pressure of the various blowing agents in Board #2 over the same time period.



**Figure 6:** Predicted 15 year thermal resistance for laminate foam aged at 73°F (23°C) - Board #2.

The 15-year time weighted average LTTR along with the 5-year instantaneous R-value were obtained from the

aging curve in Figure 6. These values are compared with the actual R-value of the foam aged in unregulated ambient conditions in the laboratory for 4.5 years. An additional 4 months of aging will be complete by the time of presentation of this paper and will be reported. The results shown in Table 4 clearly suggest that for the Board #2, the mathematical modeling and calculation algorithms developed by Huntsman Polyurethanes match the measured actual thermal resistance of pentane blown PIR laminate boards aged in an uncontrolled ambient laboratory environment for 4.5 years.



**Figure 7:** Predicted 15 year change in blowing agent partial pressure at 73°F (23°C) - Board #2.

Also the 15-year time-weighted average R-value is equivalent to the predicted R-value after 5 years.

Method	R-value (ft².h.°F/Btu.in)
5 year predicted	5.86
15 year weighted average - predicted	5.83
4.5 year measured	5.85

It can be seen from Figures 6 & 7 that the further decrease in thermal resistance in the period 15 to 50 years would be very small. This is due to the fact that the laminate is already saturated with air and the rate of loss of physical blowing agent is extremely slow.

As stated earlier, the samples used in this study were experimental products made expressly for the purpose of conducting scientific evaluations. Thus, the S770 numbers quoted in Tables 2 and 4 should not be considered typical of commercial products. The Polyisocyanurate Insulation Manufacturers Association (PIMA) has recently published average LTTR values (independently selected and third-party CAN/ULC-S770 tested) for Canadian-made polyiso

foam insulation containing hydrocarbon blowing agents [15]. These LTTR values range from 6.0 to 6.25 ft<sup>2</sup>.hr.<sup>o</sup>F/Btu.in for 1" to 4" thick product.

## CONCLUSIONS

This paper provides validation that for the boards tested in this study, the CAN ULC S770-00 method, which takes only weeks to complete, accurately predicts the foam thermal resistance after almost five years of aging in a laboratory environment. It was also shown in this paper that a 15-year time-weighted average R-value is equivalent to the actual R-value after 5 years.

For an experimental 1.5" thick pentane blown PIR board, the method predicts an LTTR well within 1% of the actual R-value after 4.5 years of ambient aging. The difference in results between the accelerated laboratory method and the real-time aging are within the proposed precision of the S770 Method.

The effective diffusion of blowing agents through a 1 mm thick layer of skin and facer are estimated here to be between 23 and 12 times slower than the respective diffusion through the core foam. This clearly suggests that the foam skin provides a greater barrier to diffusion than core foam. Thus, the S770 Method which accounts for the contribution of the skin and facer is a more precise method to determine LTTR of PIR roofing boards than the ASTM C 1303 Method.

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## **BIOGRAPHIES**

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Sachchida joined Huntsman Polyurethanes in 1987 after receiving his doctoral degree in Materials Science and Engineering from MIT. He has worked in a wide variety of application areas of polyurethane. He is currently a PU Application Development Associate in the Construction Group of the Huntsman Polyurethanes Rigid Foam Regional Development Center in West Deptford, NJ.

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Monica is a senior Technical Specialist at Huntsman Polyurethanes. Since joining in 1997, she has provided technical service and development for the door and water heater industry, and is currently providing technical service to the boardstock industry. She has a Master's degree in Chemical Engineering from Howard University, and a Bachelors degree in Chemical Engineering from Texas A & M University.

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Kristof joined Huntsman Polyurethanes in 1998 after receiving his Ph.D. degree in Polymer Chemistry from the University of Leuven - Belgium (KUL). He has been working on various fundamental issues of rigid foam such as lambda aging simulations. He is currently a project leader within the Rigid Foam Core Technology Team and is based at Everberg.