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OAK RIDGE NATIONAL LABORATORY

LOCKHEED MARTIN

Metals and Ceramics Division

INTERLABORATORY COMPARISON ON ESTIMATING THE LONG-TERM THERMAL RESISTANCE OF UNFACED, RIGID, CLOSED-CELL, POLYISOCYANURATE (PIR) FOAM INSULATION – A COOPERATIVE INDUSTRY/GOVERNMENT PROJECT

R. S. Graves, D. L. McElroy, F. J. Weaver
and D. W. Yarbroug

Date Published: January, 1995

NOTICE:

This document contains information of a preliminary nature. It is subject to revision or correction and therefore does not represent a final report.

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FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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EDITOR'S NOTE

Although ORNL has a policy of reporting its work in SI metric units, this report uses English units. The justification is that the insulation industry at present operates completely with English units, and reporting otherwise would lose meaning to the intended readership. To assist the reader in obtaining SI equivalents, these are listed below for the units occurring in this report.

Property	Unit Used	SI Equivalent
Dimension	in.	25.4 mm
Dimension	ft	0.3048 m
Density	lb/ft^3	$16.02\ kg/m^3$
Power	Btu/h	0.2929 W
Thermal conductivity	$Btu{\cdot}in./h{\cdot}ft^2{\cdot}°F$	$0.1441\ W/m{\cdot}K$
Thermal resistance	$h{\cdot}ft^2{\cdot}°F/Btu$	$0.1762\ K{\cdot}m^2/W$
Temperature	°F	°C = (5/9)(°F-32)
Temperature difference	°F	°C = (5/9)°F

ABSTRACT

The current report describes the results obtained from 13 participating laboratories in an interlaboratory comparison on a procedure to estimate the long-term thermal resistance of unfaced, rigid, closed-cell polyisocyanurate foam insulation. The procedure used ASTM Test Method C 518 for periodic determinations of the thermal resistance of specimens of reduced thickness for a period of 200 days to observe the effects of aging. The set of thermal resistance results for specimens aged under controlled laboratory conditions was used in a computer program to predict the lifetime thermal resistivity (R/inch) of the foam product under actual use conditions .

The specimens were prepared from the center of a full-thickness section in order to have a cell gas content that is representative of the section at the time of manufacture. The material for this interlaboratory comparison was a 2-inch thick polyisocyanurate boardstock blown with hydrochlorofluorocarbon 141b and CO_2 (for convenience, in the text this combination of gases is designated HCFC-141b). Five thermal resistance specimens were prepared for each participant: a full thickness specimen, 1.3 inches thick, and four thin specimens, 0.3 inches thick. Each thermal resistance test was done at a mean temperature of 75 $\pm$ 4°F on specimens conditioned at 75 $\pm$ 4°F and 50 $\pm$ 5 percent relative humidity before and during the test sequence. Ten data points spanning an aging period of 200 days were obtained for the thick specimen and for the stack of four thin specimens.

The nonlinear increase in k with time/(thickness)^2 can be described by two linear regions if one plots the natural logarithm of k versus (time)$^{1/2}$/thickness. The data are fitted using a least squares method to a straight line for region 1 and region 2. This procedure assumes the thermal conductivity (k) can be described by an exponential dependence on diffusion coefficient (D), time (t) and thickness (h).

$$Y = A + BZ \tag{1}$$

$$\ln k = \ln k_0 + (Dt)^{1/2}/h \tag{2}$$

where k_0 is the initial thermal conductivity, A is ln k_0, Y is ln k, Z is $t^{1/2}/h$, and B is $D^{1/2}$. The fitted equations for region 1 and region 2 are used to estimate k for a foam of a specific thickness at various times and the time-averaged lifetime thermal resistivity (1/k). For the Set 1 boards (test boards prepared on November 29, 1993) seven data sets had one standard deviation of 1.1% for 10-year lifetimes and 1.3% for 20-year lifetimes for the ratio of predicted lifetime R/inch value to initial R/inch value for 1.5-inch thickness. For Set 2 boards (test boards prepared on January 3, 1994 that showed significant ageing was occurring in the specimens prepared after November 29, 1993) the corresponding standard deviations for eight data sets were much greater (6.0% for 10 years and 6.6% for 20 years) due to the variation in initial R/inch values. These two data sets show that all test specimens of reduced thickness should be prepared at the same time in order to limit uncertainty in predictions.

The results of this study provide a precision and bias statement that is needed for the as-tested procedure and for an ASTM C16 standard on estimating aging effects on k.

INTRODUCTION

In April of 1993, an extension to Cooperative Research and Development Agreement (CRADA) ORNL 90-0028 was signed between the Society of Plastics Industry (SPI)-Polyurethane Division, the Polyisocyanurate Insulation Manufacturers Association (PIMA), and the Oak Ridge National Laboratory (ORNL). This CRADA extension has three tasks:

Task 1: Continue laboratory/field conditioning and testing of closed-cell polyisocyanurate foams blown with CFC-11 and HCFC-141b for one more year;

Task 2: Prepare a procedure for the laboratory determination of the long-term thermal resistance of polyisocyanurate closed-cell foams; and

Task 3: Develop a procedure for faced polyisocyanurate foams.

Task 1 developed from a cooperative industry/government program to determine the thermal performance of polyisocyanurate roof insulation foam boards blown with HCFC-141b and CFC-11. Apparent thermal conductivity (k) measurements were continued using the ORNL Unguarded Thin Heater Apparatus (UTHA) (1) on specimens exposed in the field in the ORNL Roof Thermal Research Apparatus (RTRA). In addition, Task 1 included continuing to make k-measurements using the ORNL UTHA and the ORNL Heat Flow Meter Apparatus (HFMA) (2) on specimens of three thicknesses sliced from the original boardstock and aged in the laboratory at 75°F and 150°F. Test results on this program, before the present CRADA extension was initiated, are reported in references 3, 4 and 5, and these ORNL reports list a large number of publications in the open literature. These test results showed two linear regions of logarithm of k with the quantity (diffusion coefficient x time)$^{1/2}$/thickness. This yielded effective diffusion coefficients for air into the foams and for blowing agent out of the foams. This thermal data could be expressed in terms of scaled time (time$^{1/2}$/thickness) to provide estimates of the thermal resistance or time-averaged thermal resistance for full thickness boards.

The latter observation and the development of a new ASTM standard, (6), (Standard Test Method for Estimating the Long-Term Change in the Thermal Resistance of Unfaced Rigid Closed Cell Plastic Foams by Slicing and Scaling Under Controlled Laboratory Conditions), led to Task 2 of the present CRADA extension. This current report describes the results of an interlaboratory comparison on a procedure to estimate this long-term change. Appendix A contains a summary of the PIMA Standard that was drafted as part of Task 2. Appendix B contains the details of a PIMA Standard for Describing R(time).

Task 3 of the current CRADA extension, which is a study of faced polyisocyanurate foams, will be the subject of a future report.

Closed-cell plastic foams have a thermal performance that changes with time. Over a relatively long time period (years), air components diffuse into the closed cells while the blowing agent that was captured in the cells during production diffuses out. These processes change the concentrations of the various gas species, which in turn change the thermal properties of the gas contained within the cells, and therefore the thermal performance of the plastic foam. The results of the previous CRADA (3-5) and the development of the ASTM standard test method (6) yielded a procedure that would accelerate the change in the thermal resistance of the closed cell plastic foams due to changes in the cell gas concentrations. The accelerated procedure is based on the assumption that a thin-slice removed from the core of the foam board will age at a rate that is proportional to the square of the ratio of the thickness of the product to that of the thin slice. For example, the thermal resistance of a 2-inch thick board after sixteen years is equal to the thermal resistance of a 0.5-inch thick specimen after 1 year $[(2/0.5)^2 = 16]$.

This accelerated procedure can be an extremely useful tool to the building community. Material producers have faced a major formulation change in their product lines due to the ban in the production of CFCs and will be facing further changes with the upcoming elimination of HCFC substitutes that were being used to replace CFCs. The short time period given to industry to comply to the ban does not give them the luxury to develop data that requires long-term tests to allow them to introduce and support their products to an accepting marketplace. Similarly, users of these products require long-term thermal performance data on closed-cell plastic foams to make informed design recommendations and purchasing decisions. This accelerated test procedure produces a prediction of the long-term thermal performance of a closed cell plastic foam. ASTM requires, and industry needs, a precision and bias statement to address the accuracy of this procedure. The test results from ORNL that were produced during the earlier CRADA showed field and laboratory data that agreed on the long-term performance of both CFC- and HCFC-blown products. This showed the procedure does have the ability to predict the long-term thermal performance with acceptable accuracy. Task 2 of the current CRADA provides test results from an extensive interlaboratory comparison as the means to address the accuracy of the prediction and to obtain the required precision and bias statement for the procedure.

EQUIPMENT

Thirteen laboratories, listed in Table 1, participated in this interlaboratory comparison of the procedure to estimate the long-term thermal performance of polyisocyanurate (PIR) foam insulation. Three different sizes (12, 18, and 24 inch-square specimen openings) of Heat Flow Meter Apparatuses (HFMAs) were used. Each of these HFMAs is capable of measuring k in accordance with the ASTM C 518 Test Method (2). The characteristics of these HFMAs are listed in Table 2. The ORNL HFMA, which is the baseline apparatus for the previous CRADA is described elsewhere (7). For the current CRADA, ORNL used two HFMAs produced by LaserComp. The LaserComp Fox 300 can accommodate a 12 x 12 inch square specimen and has a 3 x 3 inch heat flux transducer (HFT) in each of its constant temperature plates. The LaserComp Fox 600 can accommodate a 24 x 24 inch square specimen and has an array of 16 HFTs, each 4 x 4 inches, in each of its constant temperature plates.

Table 1. Laboratories participating in the interlaboratory comparison of the procedure to estimate the long-term thermal performance of PIR foam insulation.

Participant
Allied Signal, Inc.
Atlas Roofing Corporation
BASF Corporation
Center for Applied Engineering
Dow Chemical Co.
Firestone Building Products
Holometrix, Inc.
ICI Polyurethanes
LaserComp, Inc.
Miles, Inc.
NRG Barriers, Inc.
National Institute of Standards and Technology
Oak Ridge National Laboratory

Table 2. Characteristics of HFMAs used in the interlaboratory comparison of the procedure to estimate the long-term performance of PIR foam insulation.

PART.	APPARATUS	SIZE, inches	HFT SIZE, inches	CAL. STD.
A	Dynatech R/D, K-matic	12 x 12	4 x 4	Fiberglass
B	Holometrix,Inc., K-matic	12 x 12	4 x 4	SRM 1450b
C	Holometrix,Inc., K-matic	12 x 12	4 x 4	SRM 1450b
D	Holometrix,Inc., Rapid-k	12 x 12	4 x 4	SRM 1450b
E	Holometrix,Inc., Rapid-k	12 x 12	4 x 4	Fiberglass
F	Holometrix,Inc., Rapid-k	12 x 12	4 x 4	SRM 1450b
G	Holometrix,Inc., Rapid-k	12 x 12	4 x 4	SRM 1450b
H	Holometrix,Inc., K-matic	12 x 12	4 x 4	SRM 1450b
I	LaserComp, Fox 300	12 x 12	3 x 3	SRM 1450b
J	Holometrix,Inc., K-matic	12 x 12	4 x 4	SRM 1449 SRM 1450b
K	Sparrel Engr., TC-100	18 x 18	6 x 6	Fiberglass
L	Holometrix,Inc., Rapid-k	12 x 12	6 x 6	SRM 1450b
M-1	LaserComp, Fox 300	12 x 12	3 x 3	SRM 1450b
M-2	LaserComp, Fox 300	12 x 12	3 x 3	SRM 1450b
M-3	LaserComp, Fox 600	24 x 24	16:4 x 4 array	SRM 1450b

Five participants operate K-matic HFMAs produced by Dynatech R/D or Holometrix, Inc. having 12 x 12 inch square specimen openings and 4 x 4 inch square HFTs in only one bounding surface. Five participants operate Rapid-k HFMAs produced by Holometrix, Inc. having 12 x 12 inch square specimen openings and 4 x 4 inch square or 6 x 6 inch square HFTs in only one bounding surface. One participant, other than ORNL, used a LaserComp Fox 300 HFMA having a 12 x 12 inch specimen opening and a 3 x 3 inch HFT in each of its constant temperature plates. One participant used a TC-100 HFMA produced by Sparrel Engineering having a 18 x 18 inch specimen opening and a 6 x 6 inch HFT. ORNL used Fox 300 (3 x 3 inch HFTs) and Fox 600 (4 x 4 array) HFMAs.

A HFMA that operates in accordance to ASTM C 518 (2) establishes a steady-state, unidirectional heat flow through a test specimen placed between two parallel plates at constant, but different temperatures. Normally, heat flux transducers (HFT) are mounted in one or both of the two plates and are calibrated using standards in accordance with ASTM C 1132 (8) by the equation

$$S = \frac{k\{T(h) - T(c)\}}{V\,L} \qquad (3)$$

where S is the HFT sensitivity in $(Btu/h \cdot ft^2)$-V, k is the apparent thermal conductivity of the standard in $(Btu \cdot in/h \cdot ft^2 \cdot °F)$, T(h) and T(c) are the temperatures of the two plates in °F, L is the thickness of the standard in inches, and V is the output of the HFT in V. Each of the participants used calibration standards of either fiberglass (SRM 1450b) or fumed silica (SRM 1449).

The quantities measured during a routine test of a specimen with an unknown k are the same as those measured during a calibration. The k of the unknown is computed from

$$k = \frac{(S\,V)\,L}{\{T(h) - T(c)\}} \qquad (4)$$

For Eq. 4 to be valid strictly, the test specimen must be homogeneous, as is required by ASTM C 518. In performing part of the interlaboratory comparison, however, it was necessary to place some of the specimens in a "picture frame" constructed from high density fiberglass insulation. This type of composite specimen was used at ORNL on the 18 x 18 inch square specimens similar to those tested by Participant K. This type of composite specimen reduces extraneous heat losses from the edges of the specimens.

SPECIMENS

Test specimens for this interlaboratory comparison were produced from polyisocyanurate boardstock that was produced by Atlas Roofing Corporation at their plant in East Moline, Illinois on November 1, 1993. The experimental, non-commercial laminate boardstock was blown with hydrochlorfluorocarbon-141B and CO_2. (For convenience in this report—text, figures, and tables—this combination of blowing gases is designated HCFC-141b.) This boardstock had a GAF black felt facer and the as-manufactured thickness was nominally 2 inches. The boardstock arrived at ORNL on November 19, 1993 and two sets of test specimens were planed or sliced from this boardstock at ORNL. Each set included five slices: one thick board, nominally 1.3-inches thick, and four thin boards, each nominally 0.3-inches thick. Set 1 was produced on November 29, 1993 and Set 2 was produced on January 3, 1994. The specimen size was either 12 x 12-inches square or 18 x 18-inches square. The 12 x 12-inch test specimens were sliced from the boardstock and the 18 x 18-inch test specimens were planed from the boardstock. Material was planned from each face, so each of the 1.3- and 0.3-inch thick test specimens contained the boardstock centerline. Annex E contains a discussion of test specimen preparation procedures.

Before the sets of test specimens were distributed by ORNL to the participants, measurements were made at ORNL of thicknesses, weights, and apparent thermal conductivities of the test specimens. The thickness for the thick boards and the stack of four thin specimens was determined in the ORNL HFMAs. This provided each participant with a starting test thickness. The weights of the thick specimens were measured to 0.01 grams using an electronic laboratory balance. The apparent thermal conductivity of each of the thick test specimens was determined before the two sets of test specimens were shipped to the participants. Table 3 indicates the participants that received Set 1 (time = 0 is November 29, 1993) and Set 2 (time = 0 is January 3, 1994).

Table 3. Participants that received Set 1 and Set 2 of the test specimens for the interlaboratory comparison.*

Set 1: Time = 0 is November 29, 1993.	Set 2: Time = 0 is January 3, 1994.
Shipped by ORNL on December 3, 1993.	Shipped by ORNL on January 5, 1994.
ICI, DOW, Firestone, Allied, Atlas, Holometrix, and ORNL-1.	Miles, NRG, NIST, CAE, BASF, LaserComp, ORNL-2, and ORNL-3.

*Test results were received at ORNL in August and September 1994.

Table 4 contains the first measured apparent thermal conductivity test result obtained by ORNL and the first measured k-value obtained by the participants on their thick board. The average of the ORNL test results was 0.1387 $Btu{\cdot}in./h{\cdot}ft^2{\cdot}°F$. The ORNL k-values ranged from 0.1324 to 0.1545 $Btu{\cdot}in./h{\cdot}ft^2{\cdot}°F$, or more than 16%. This was a surprisingly large spread in values. The percent differences in these data are given in Table 4. The values for the individual boards agree to better than 3.3% and the average of the percent differences was 1.1%. Since these individual thick test specimens are aging with time (k is increasing with time), and since the measurements of k at ORNL were performed before the measurements by the participants, it is especially noteworthy that the k results from ORNL are lower than the k-results by the participants in 9 of 11 cases.

Table 4. The first measured apparent thermal conductivity test results obtained on the thick boards by ORNL and the first measured apparent thermal conductivity test results by the participants.

Set.Board Numbers Thick/Thin	First Measured k-Value on Thick Board by Participant (Btu·in./h·ft^2·°F)	First Measured k-Value on Thick Board by ORNL (Btu·in./h·ft^2·°F)	Percent Differ, %
A. 3 / 18, 19, 20, 21	0.1360	0.1332	+2.1
B. 1 / 10, 11, 12, 13	0.1382	0.1338	+3.3
C. 49 / 75, 76, 77, 78	0.1370	0.1355	+1.1
D. 47 / 67, 68, 69, 70	0.1470	0.1452	+1.9
E. 6 / 30, 31, 32, 33	0.1370	0.1328	+3.2
F. 5 / 26, 27, 28, 29	0.1336	0.1327	+0.7
G. 2 / 14, 15, 16, 17	0.1338	0.1332	+0.5
H. 4 / 22, 23, 24, 25	0.1360	0.1324	+2.7
I. 45 / 56, 58, 59, 61	0.1421	0.1393	+2.0
J. 48 / 71, 72, 73, 74	0.1370	0.1406	-2.5
K. 83 / 87, 88, 91, 92	0.1560	not measured	
L. 46 / 63, 64, 65, 66	0.1496	0.1545	-3.2
M-1. 7 / 34, 35, 36, 37	0.1317	0.1317	
M-2. 50 / 55, 57, 60, 80	0.1455	0.1455	
M-3. 82 / 85, 86, 89, 90	0.1389	0.1389	

PROCEDURE

Each participant received a copy of the PROCEDURE FOR INTERLABORATORY COMPARISON (which is given in Appendix C of the current report), the five pieces of sliced PIR foam, and a program diskette for recording and analyzing their data. The PROCEDURE contains 15 statements, including specimen descriptions, data to be provided by ORNL, request for HFMA calibration, suggested data on each specimen to be obtained by the participant, stacking and testing times for the test specimens, reporting procedures, and data analysis examples. These instructions asked the participants to perform twenty apparent thermal conductivity measurements on the specimen sets as they aged from 2 to 200 days at 75°F. Doing this would provide 10 data points on the thick specimens and 10 data points on the stack of four thin specimens. The participants were asked to complete their testing in July 1994 and to provide their data sets to ORNL in August and September 1994. Most of the participants did so.

RESULTS

The k measurements obtained in the laboratory intercomparison are listed by participant in the tables contained in Appendix D. Data analysis was performed at ORNL on the data obtained by each participant in the following manner:

Step 1. The individual data sets were entered in a LOTUS 1-2-3 spreadsheet, which yielded each multicolumn table given in Appendix D. Properties calculated on this data sheet included the "X" variable which is the scaled time-thickness variable of $(\text{time})^{1/2}$/thickness in the units of $(\text{days})^{1/2}$/inch and the "Y" variable which is ln 100·k with k in the units of $\text{Btu·in./(h·ft}^2\text{·°F)}$.

Step 2. The individual data sets were sorted in ascending order of the "X" variable which is $(\text{time})^{1/2}$ / thickness.

Step 3. The individual data sets were plotted as "Y" versus "X", or "ln 100·k" versus "$\text{days}^{1/2}$ / inch". Doing so resulted in a "two-region" graph with Region 1 being the initial low k, high slope region and Region 2 being the high k, low slope region. Figures 1, 2, 3, and 4 are examples of these plots and these are discussed below. Appendix D contains similar plots for each individual data set.

Step 4. The individual data points in Region 1 and the individual data points in Region 2 were identified. This process often yielded one or two data points that were in the transition range and such data points were not included in the analyses.

Step 5. The Region 1 and Region 2 data were fitted by means of a least squares method to the following functions:

$$\text{Region 1: } \ln 100\cdot k = A_1 + B_1\,(\text{days})^{1/2} / \text{inch} \qquad (5)$$

$$\text{Region 2: } \ln 100\cdot k = A_2 + B_2\,(\text{days})^{1/2} / \text{inch} \qquad (6)$$

Appendix D contains graphs for data from each participant and the two regions are readily seen in each graph. The results of the fitting by means of a least squares method are on each graph.

Step 6. For each data set, the constants (A_1 and A_2) and slopes (B_1 and B_2) for Region 1 and Region 2 were used to calculate the average k for 10 years with 1.5 and 2.0 inches for the product thickness and 20 years with 1.5 and 2.0 inches for the product thickness. This calculation yielded the predicted lifetime R/inch values.

Step 7. For each data set the Region 1 constant, A_1, was used to calculate the predicted initial k value:

$$A = \ln 100 \cdot k_0 \tag{7}$$

where k_0 is the initial predicted k-value.

As an example of this data analysis, Figure 1 shows the results obtained by ORNL on Board 7 (thick), and Boards 34, 35, 36 and 37 (stacked, thin). These boards were part of the Set 1 boards.

Region 1 contains 17 individual data points and Region 2 contains 12 individual data points. One data point is in the transition range. Figure 2 shows the results obtained by ORNL on Board 50 (thick), and Boards 55, 57, 60 and 80 (stacked, thin). These boards were part of the Set 2 boards. Region 1 contains 17 individual data points and Region 2 contains 10 individual data points. Two data points are in the transition range. The results given in these two figures show the two regions represented by Region 1 and Region 2.

The fitting procedure yielded the values for A and B for Region 1 and Region 2 given in Table 5.

Table 5. Values for constants and slopes for Region 1 and Region 2 for ORNL data on boards from Set 1 and Set 2.

	Constants for Set 1*	Slopes for Set 1*	Constants for Set 2**	Slopes for Set 2**
Region 1	2.5422	0.03000	2.6545	0.02203
Region 2	2.8717	0.002885	2.9227	0.001759

* Set 1 boards were 7, 34, 35, 36 and 37
**Set 2 boards were 50, 55, 57, 60 and 80

PIMA RR (HCFC-141b)
BOARDS # 7/34, 35, 36, 37.

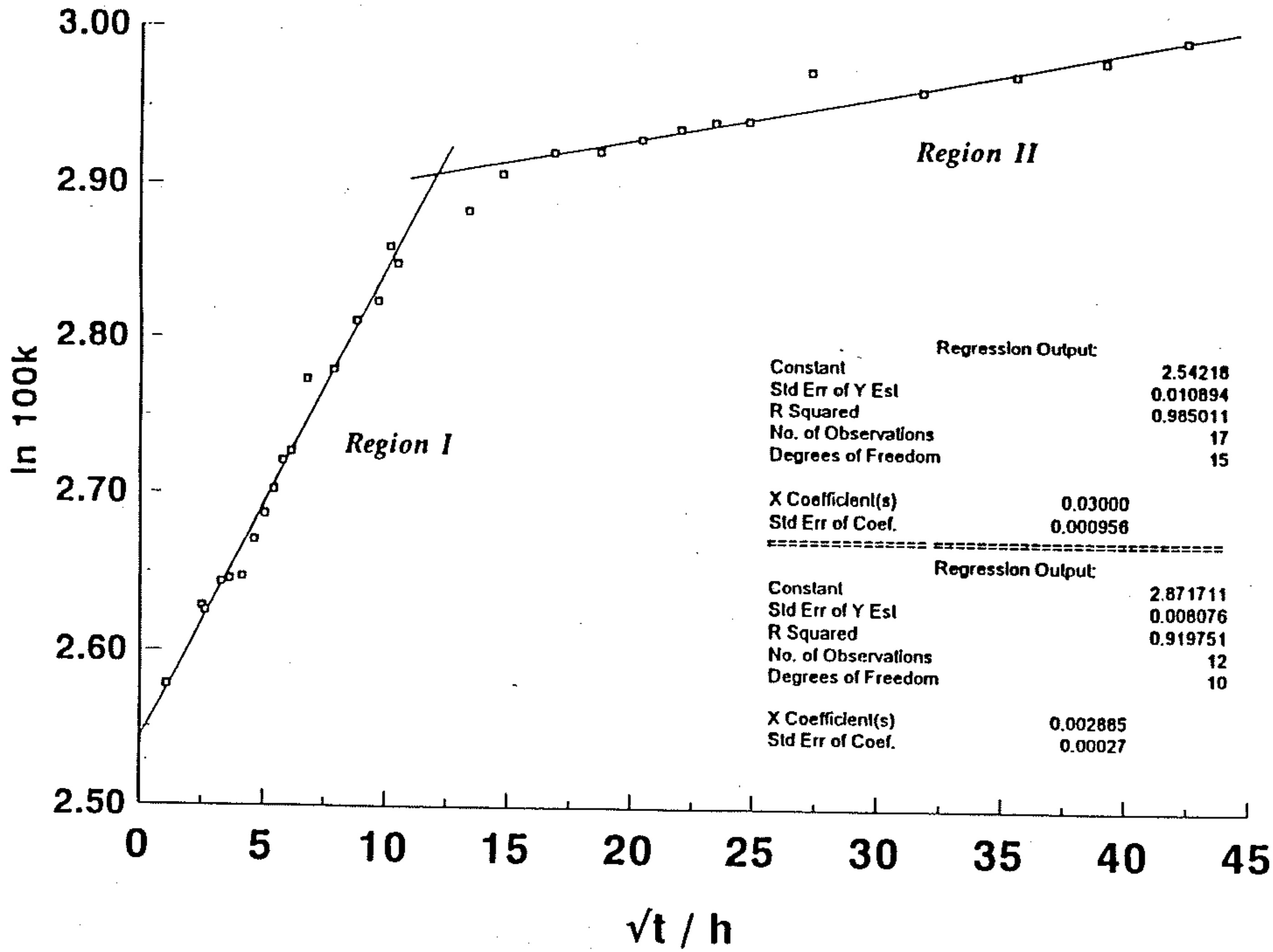

Figure 1. Results obtained on Set 1 boards 7, 34, 35, 36, and 37.

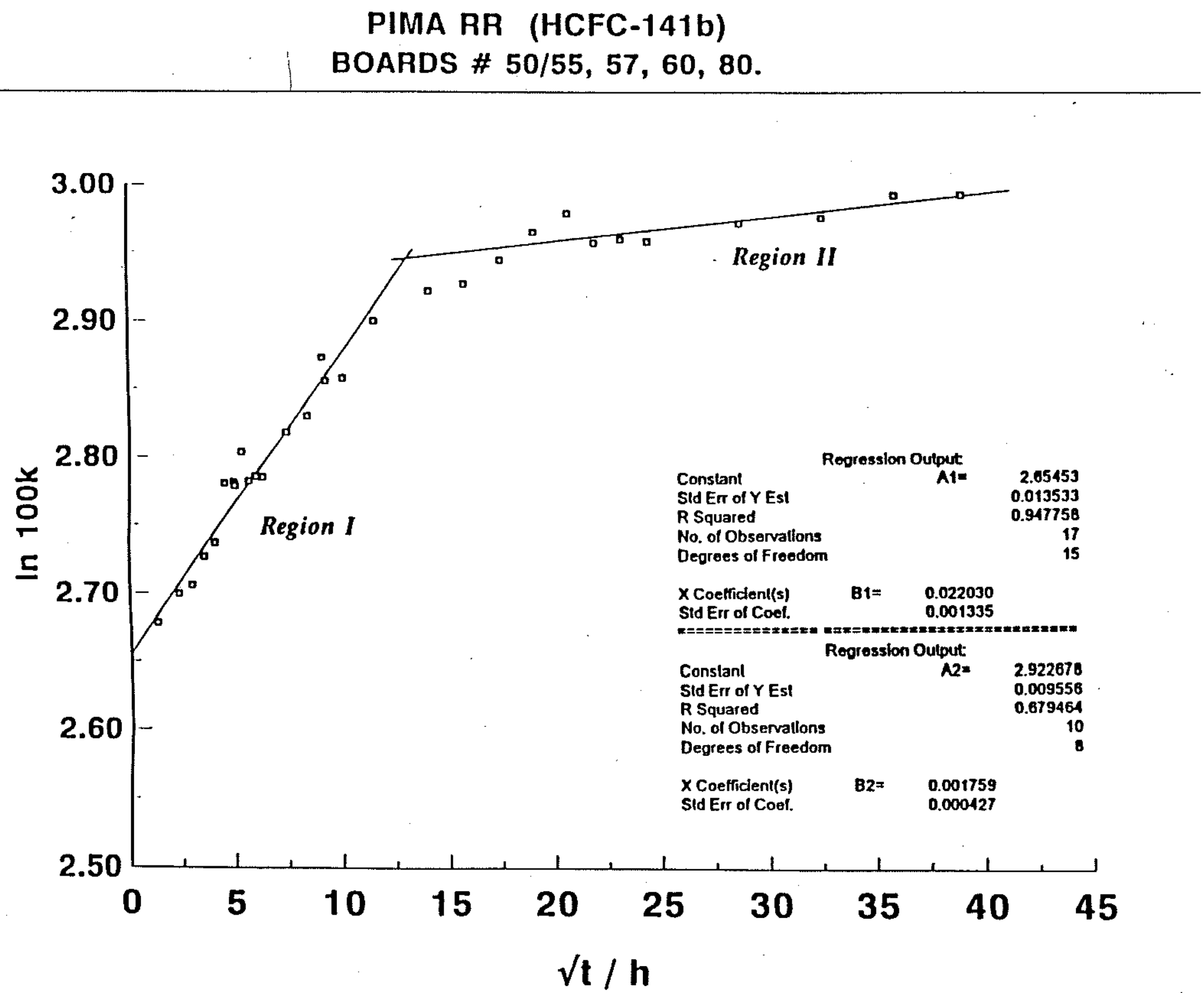

Figure 2. Results obtained on Set 2 boards 50, 55, 57, 60, and 80.

comparisons of these two figures and the resulting data fits show the following:

First, for Set 1 boards the constant is lower for Region 1 than for Region 2 and the slope for Region 1 is about 10 times greater than the slope for Region 2. Second, for the Set 2 boards a similar comparison can be made for the constants of Region 1 and Region 2 and for the slopes of Region 1 and Region 2. The lower constants for Region 1 are associated with the lower initial thermal conductivity values. The higher slopes for Region 1 are associated with a greater diffusion coefficient for air-components than for the blowing agent (HCFC-141b). As expected an overlay of Figure 1 and Figure 2 shows the same effect that the data fits show. One very noteworthy effect that can be observed is that the constant for Region 1 for Set 1 is lower than the constant for Region 1 for Set 2. This is interpreted to mean that the boardstock aged after Set 1 was produced and before Set 2 was produced. If the boardstock had not aged during this period, November 29, 1993 to January 3, 1994, one would have expected the Region 1 constants and slopes for Set 1 and Set 2 to have agreed.

Figure 3 shows the results obtained by participant A on Set 1 boards 3, 18, 19, 20, and 21. Figure 4 shows the results obtained by participant I on Set 2 boards 45, 56, 58, 59, and 61. These two figures and the resulting constants and slopes given in Table 6 for the data fits show that the individual participants observed differences in Set 1 and Set 2 boards that were very similar to the observations made from the ORNL data sets.

Table 6. Values for constants and slopes for Region 1 and Region 2 for data on boards from Set 1 and Set 2 boards from two participants.

	Constants for Set 1*	Slopes for Set 1*	Constants for Set 2**	Slopes for Set 2**
Region 1	2.5243	0.03136	2.5941	0.02573
Region 2	2.8905	0.001825	2.9187	0.001890

* Set 1 boards were 3, 18, 19, 20 and 21 tested by participant A.
**Set 2 boards were 45, 56, 58, 59 and 61 tested by participant I.

Tables 7 and 8 contain the results of fitting the individual data sets with a least squares method as described in Step 5. The constants and slopes given in Tables 5 and 6 are repeated in Tables 7 and 8. In addition Table 7 (Region 1) includes, the r^2-value for the individual fits for Region 1; the constants and slopes for a total fit on 15 data sets using 189 data points; and a total fit on 7 data sets that represent Set 1 boards only using 83 points. Table 8 (Region 2) includes similar quantities, i.e., the r^2-value for the individual fits for Region 2; the constants and slopes for a total fit on 15 data sets using 115 data points; and a total fit on 7 data sets that represent Set 1 boards only using 55 points.

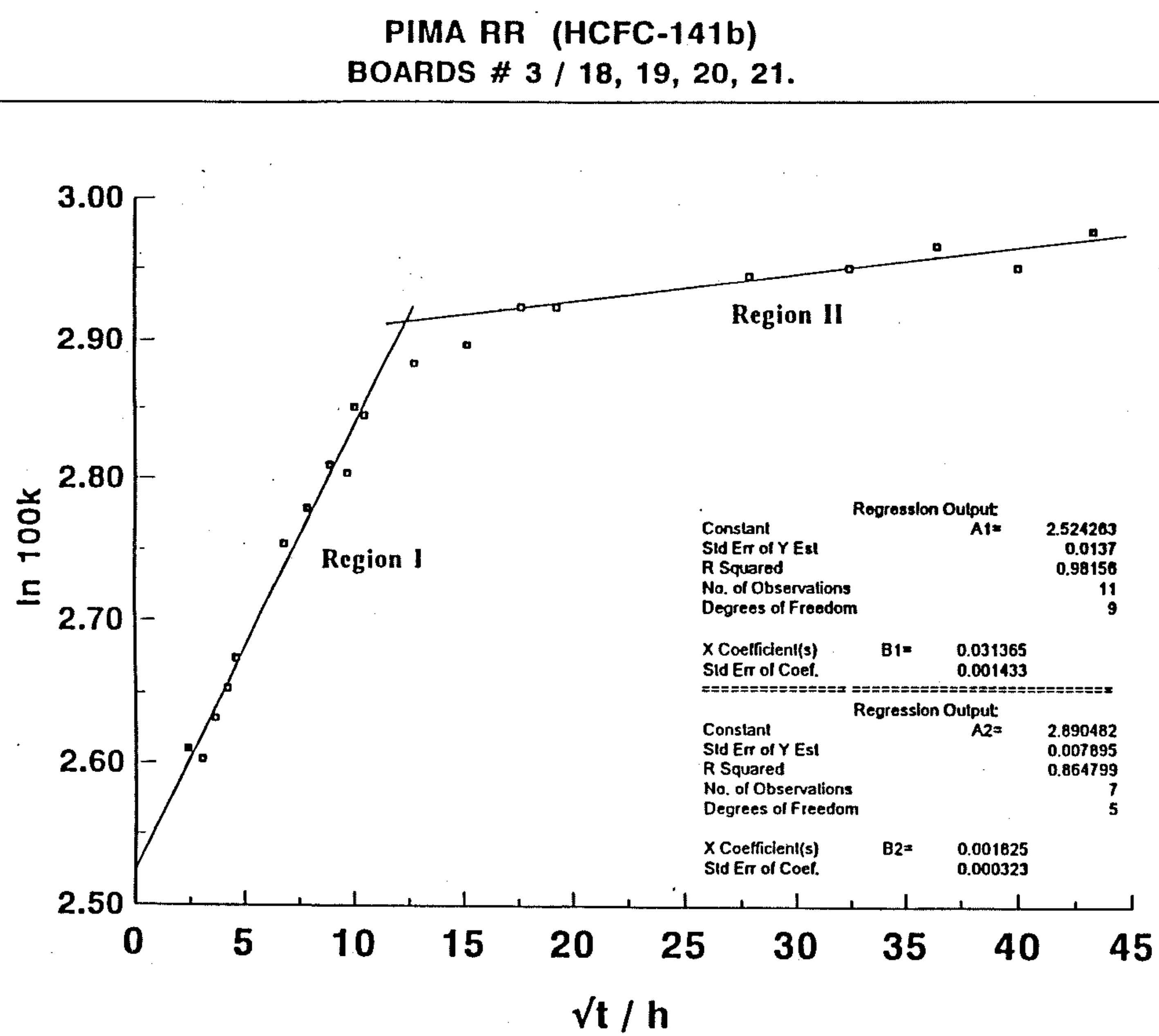

Figure 3. Results obtained on Set 1 boards 3, 18, 19, 20, and 21.

Figure 3. Results obtained on Set 1 boards 3, 18, 19, 20, and 21.

PIMA RR (HCFC-141b)
BOARDS # 45 / 56, 58, 59, 61.

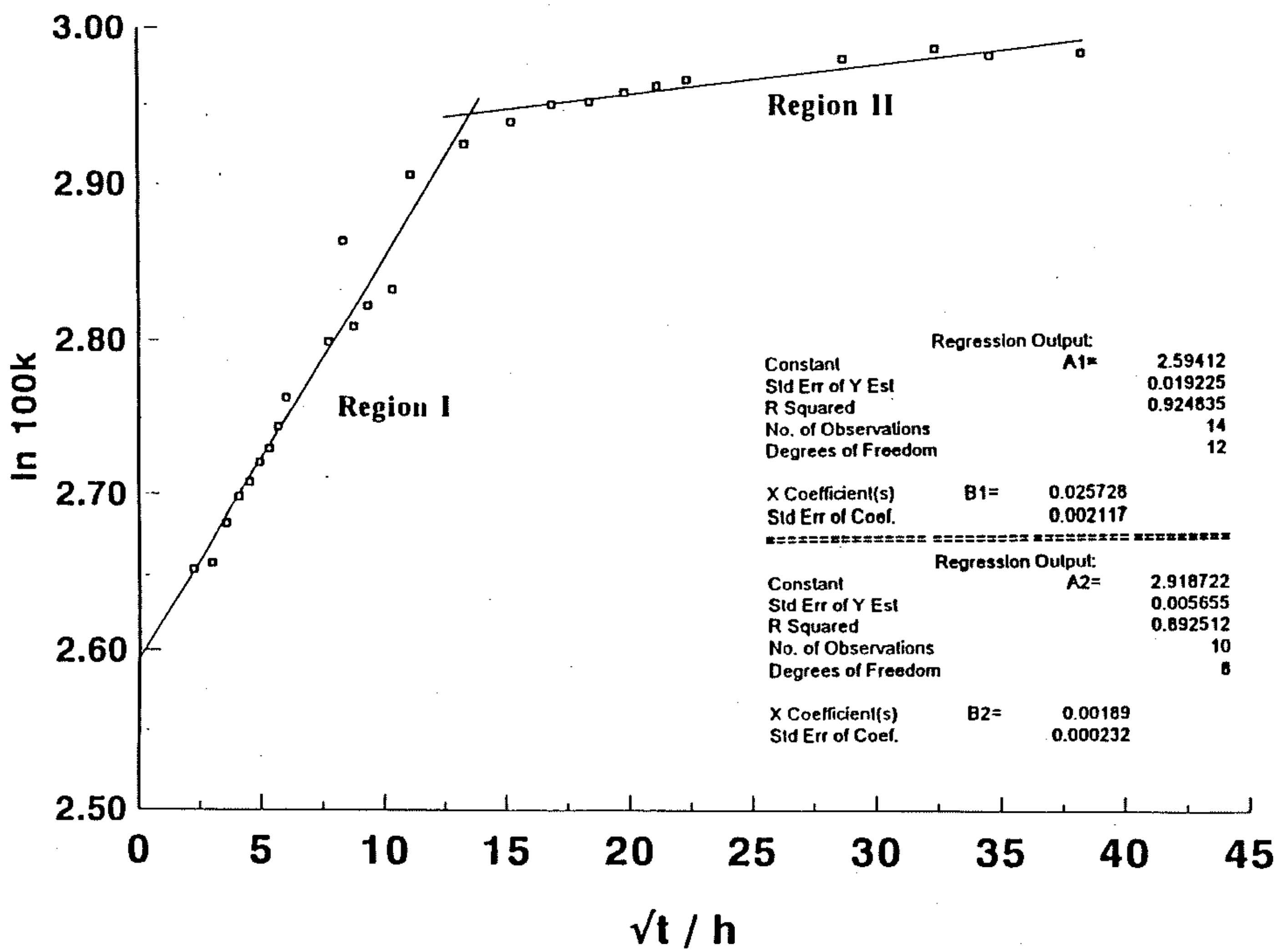

Figure 4. Results obtained on Set 2 boards 45, 56, 58, 59, and 61.

Table 7. Constants, Slopes, and r^2-values for fitting Region 1 data by means of a least squares method.

Set. Board Numbers: Thick/Thin	Region 1 Constant A	Region 1 Slope B	Region 1 r^2-Value
A. 3 / 18, 19, 20, 21	2.5243	0.03136	0.98
B. 1 / 10, 11, 12, 13	2.5407	0.02984	0.99
C. 49 / 75, 76, 77, 78	2.5626	0.02751	0.90
D. 47 / 67, 68, 69, 70	2.6349	0.02104	0.90
E. 6 / 30, 31, 32, 33	2.5333	0.02908	0.96
F. 5 / 26, 27, 28, 29	2.5311	0.03065	0.97
G. 2 / 14, 15, 16, 17	2.5481	0.02813	0.96
H. 4 / 22, 23, 24, 25	2.5311	0.03266	0.97
I. 45 / 56, 58, 59, 61	2.5941	0.02573	0.92
J. 48 / 71, 72, 73, 74	2.5947	0.02477	0.88
K. 83 / 87, 88, 91, 92	2.7183	0.01642	0.98
L. 46 / 63, 64, 65, 66	2.6689	0.02132	0.98
M-1. 7 / 34, 35, 36, 37	2.5422	0.02999	0.98
M-2. 50 / 55, 57, 60, 80	2.6545	0.02203	0.95
M-3. 82 / 85, 86, 89, 90	2.5948	0.02595	0.92
Total Fit on 15 Data Sets (189 Points)	2.59297	0.025471	0.79
Total Fit on 7 Data Sets (Set 1, 83 Points)	2.53716	0.030086	0.97

The data analysis for the Region 1 data for this interlaboratory comparison shows the 15 individual data sets had r^2-values above 0.88 that ranged from 0.88 to 0.99, while all 15 data sets (189 data points) had an r^2-value of 0.79, but a total fit on 7 data sets that were on Set 1 boards had an r^2-value of 0.97. This reduction in r^2-value is believed due to the exclusion of the Set 2 boards from the treated data set and is further indication that the boardstock was aging before the Set 2 boards were produced.

Table 8. Constants, Slopes, and r^2-values for fitting Region 2 data by means of a least squares method.

Set. Board Numbers: Thick/Thin	Region 2 Constant A	Region 2 Slope B	Region 2 r^2-Value
A. 3 / 18, 19, 20, 21	2.8905	0.001824	0.86
B. 1 / 10, 11, 12, 13	2.8906	0.002482	0.96
C. 49 / 75, 76, 77, 78	2.8318	0.004518	0.92
D. 47 / 67, 68, 69, 70	2.9000	0.001349	0.73
E. 6 / 30, 31, 32, 33	2.8509	0.002529	0.96
F. 5 / 26, 27, 28, 29	2.8652	0.002245	0.92
G. 2 / 14, 15, 16, 17	2.9457	0.000906	0.44
H. 4 / 22, 23, 24, 25	2.9038	0.001527	0.91
I. 45 / 56, 58, 59, 61	2.9187	0.001890	0.89
J. 48 / 71, 72, 73, 74	2.8872	0.001890	0.57
K. 83 / 87, 88, 91, 92	2.8861	0.001522	0.94
L. 46 / 63, 64, 65, 66	2.8564	0.003337	0.95
M-1. 7 / 34, 35, 36, 37	2.8717	0.002885	0.92
M-2. 50 / 55, 57, 60, 80	2.9227	0.001759	0.88
M-3. 82 / 85, 86, 89, 90	2.9222	0.001485	0.77
Total Fit on 15 Data Sets (115 Points)	2.89183	0.002021	0.50
Total Fit on 7 Data Sets (Set 1, 55 Points)	2.88431	0.002087	0.64

The data analysis for the Region 2 data for this interlaboratory comparison shows the 15 individual data sets had r^2-values as low as 0.44 that ranged from 0.44 to 0.96, while all 15 data sets (115 data points) had an r^2-value of 0.50, but a total fit on 7 data sets using 55 data points gave Set 1 boards an r^2-value of 0.64. The r^2-values for Region 2 were lower than those for Region 1, and did not improve dramatically by treating Set 1 boards only.

Figures 5 and 6 illustrate the specific effects that are described above. Figure 5 is a plot of all of the data that was obtained in this interlaboratory comparison on Set 1 and Set 2 boards. The Region 1 data shows a range of about 0.2 units and the Region 2 data shows a range of about 0.1 units. Figure 6 shows all of the data that was obtained in this interlaboratory comparison on Set 1 boards only. Figure 6 shows much smaller data ranges for Region 1 and Region 2.

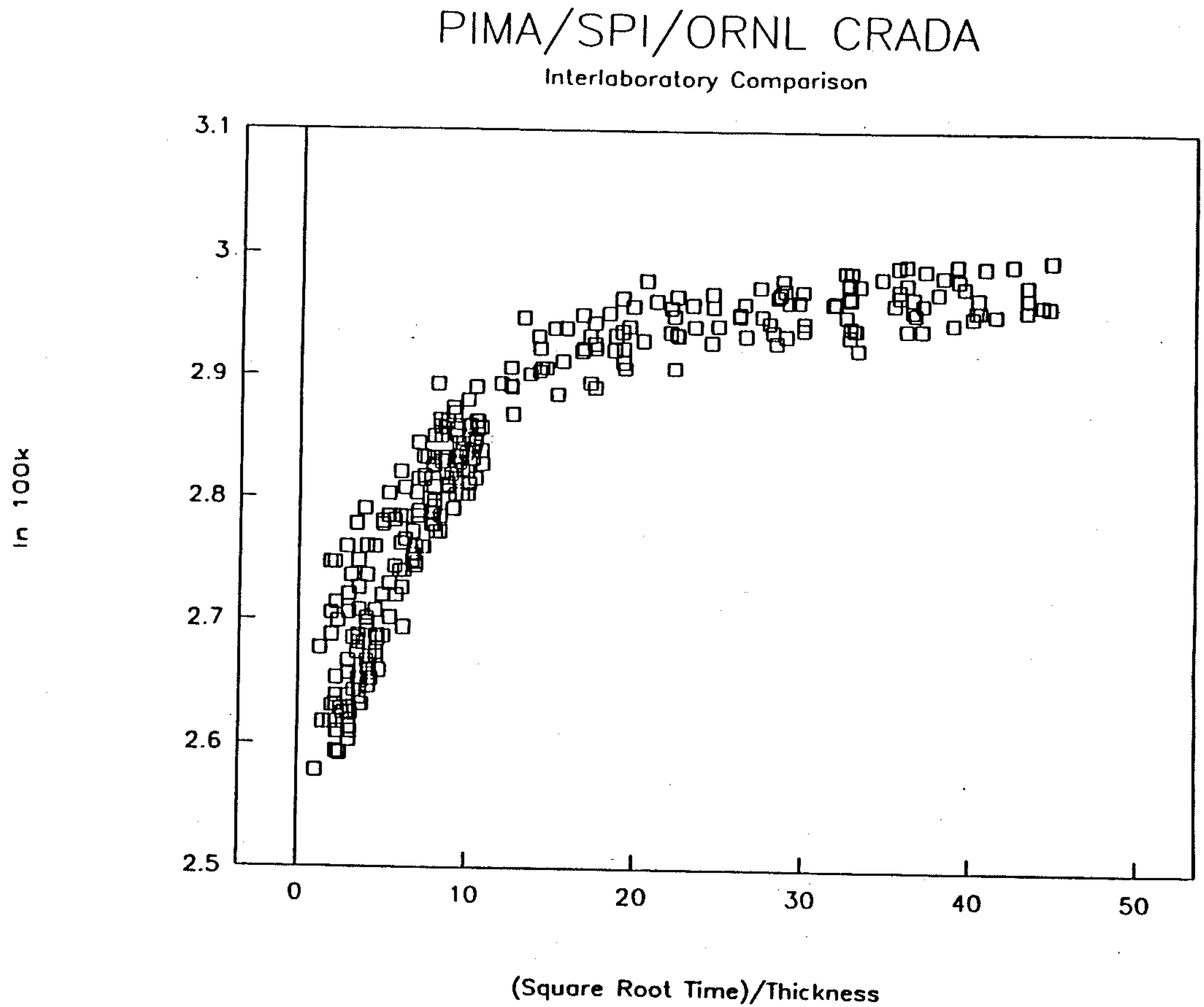

Figure 5. Complete set of results for Set 1 and Set 2 boards for the *interlaboratory comparison.*

Figure 5. Complete set of results for Set 1 and Set 2 boards for the interlaboratory comparison.

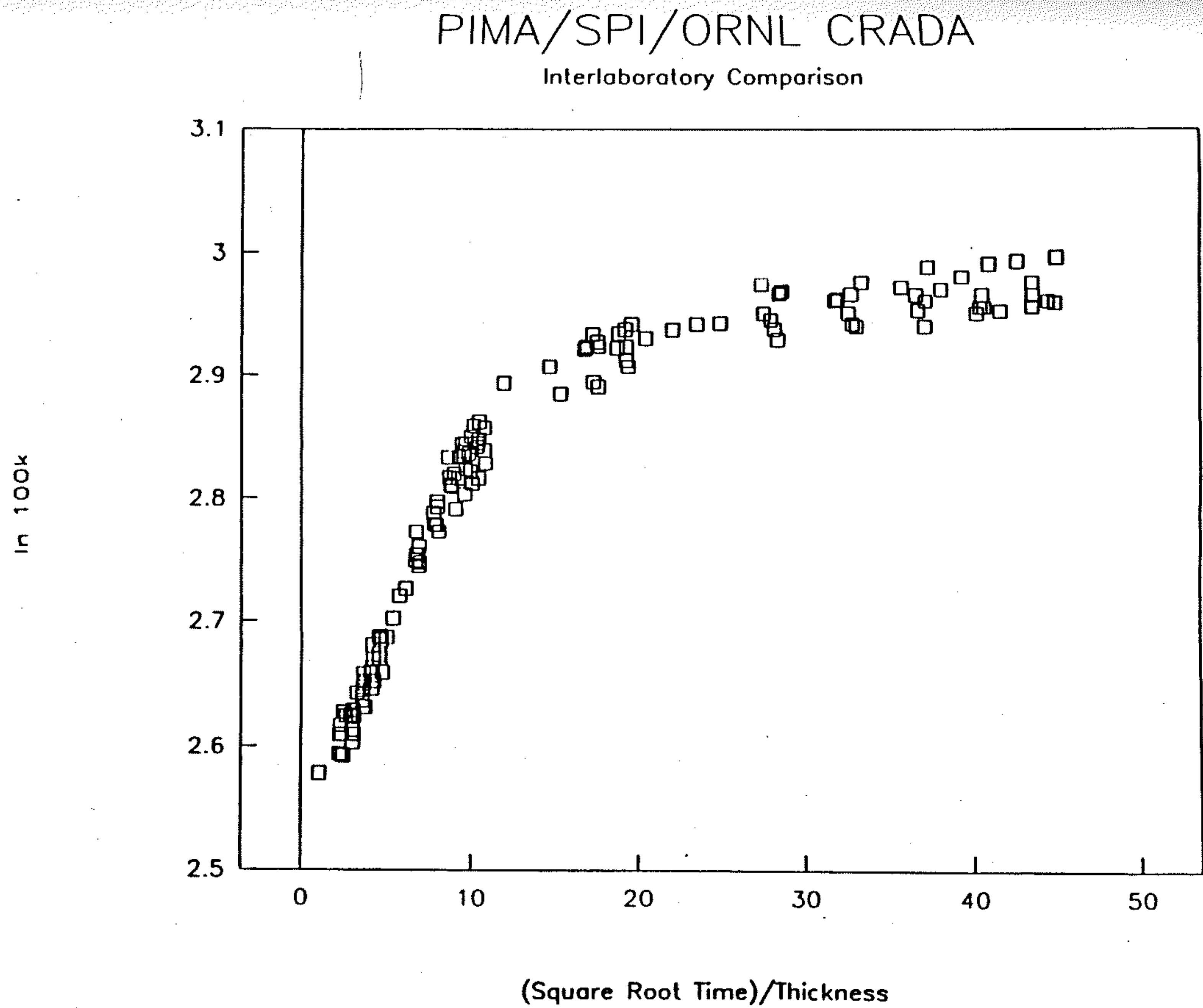

Figure 6. Set of results for Set 1 boards only for the interlaboratory comparison.

As indicated in Step 7, for each data set, the Region 1 constant, A_1, was used to calculate the predicted initial k value:

$$A = \ln 100 \cdot k_o \tag{7}$$

where k_o is the initial predicted k-value.

Table 9 contains the predicted initial k-value using a fitting equation as shown by Equation 2 for the Region 1 results. The predicted initial k-values obtained from the constant term in Region 1 had a 20% range of values, i.e., 0.1248 to 0.1515 Btu·in/(h·ft^2·°F), with a range of 0.0267 Btu·in/(h·ft^2·°F) and an average value of 0.1328 Btu·in/(h·ft^2·°F). The variance was 6.6281 x 10^{-5} and this yields a one standard deviation value of 6.1%.

Table 9. Predicted initial k-value using data fits for Region 1.

Set. Board Numbers: Thick/Thin	Predicted Initial k-Value Using Equation for Region 1 (Btu·in./h·ft^2·°F)
A. 3 / 18, 19, 20, 21	0.1248 Low
B. 1 / 10, 11, 12, 13	0.1269
C. 49 / 75, 76, 77, 78	0.1297
D. 47 / 67, 68, 69, 70	0.1394
E. 6 / 30, 31, 32, 33	0.1260
F. 5 / 26, 27, 28, 29	0.1257
G. 2 / 14, 15, 16, 17	0.1278
H. 4 / 22, 23, 24, 25	0.1257
I. 45 / 56, 58, 59, 61	0.1338
J. 48 / 71, 72, 73, 74	0.1339
K. 83 / 87, 88, 91, 92	0.1515 High
L. 46 / 63, 64, 65, 66	0.1442
M-1. 7 / 34, 35, 36, 37	0.1271
M-2. 50 / 55, 57, 60, 80	0.1422
M-3. 82 / 85, 86, 89, 90	0.1339

Because this was a surprisingly large value, the predicted initial k-value was separated for Set 1 boards and for Set 2 boards, as shown in Table 10. The predicted initial k-value obtained from the constant term in Region 1 for specimens produced on November 29, 1993 (Set 1) only had a range of 2.4%, i.e., 0.1248 to 0.1278 Btu·in/(h·ft^2·°F), with a range of 0.0030 Btu·in/(h·ft^2·°F) and an average value of 0.1263 Btu·in/(h·ft^2·°F). The variance was 1.05 x 10^{-6} and this yielded one standard deviation value of 0.8%. Table 11 summarizes this for Set 1 and Set 2 boards. This is further proof that significant aging was occurring in the boardstock after November 29, 1993 and this indicates that all test specimen production from boardstock should be done at the same time.

Table 10. Predicted initial k-value using data fits for Region 1, but separated into Set 1 boards and Set 2 boards.

Set. Board Numbers: Thick/Thin	Predicted Initial k-Value Using Equation for Region 1 (Btu·in./h·ft^2·°F) Set 1	Set 2
A. 3 / 18, 19, 20, 21	0.1248 Low	
B. 1 / 10, 11, 12, 13	0.1269	
C. 49 / 75, 76, 77, 78		0.1297 Low
D. 47 / 67, 68, 69, 70		0.1394
E. 6 / 30, 31, 32, 33	0.1260	
F. 5 / 26, 27, 28, 29	0.1257	
G. 2 / 14, 15, 16, 17	0.1278 High	
H. 4 / 22, 23, 24, 25	0.1257	
I. 45 / 56, 58, 59, 61		0.1338
J. 48 / 71, 72, 73, 74		0.1339
K. 83 / 87, 88, 91, 92		0.1515 High
L. 46 / 63, 64, 65, 66		0.1442
M-1. 7 / 34, 35, 36, 37	0.1271	
M-2. 50 / 55, 57, 60, 80		0.1422
M-3. 82 / 85, 86, 89, 90		0.1339

Table 11. Predicted initial k-values obtained from the constant term for Region 1 separated for Set 1 and Set 2 boards.

SLICING DATE	NOVEMBER 29, 1993	JANUARY 3, 1994
BOARD SET	1 (7 SETS)	2 (8 SETS)
HIGH k	0.1278	0.1515
LOW k	0.1248	0.1297
RANGE OF k	0.0030 OR 2.4%	0.0218 OR 15.7%
AVERAGE k	0.1263	0.1386
VARIANCE	1.0514×10^{-6}	5.111×10^{-5}
ONE STANDARD DEVIATION	0.8%	5.2%

As indicated in Step 6, for each data set, the constants (A_1 and A_2) and slopes (B_1 and B_2) for Region 1 and Region 2 were used to calculate the average k for 10 years with 1.5 and 2.0 inches for the product thickness and 20 years with 1.5 and 2.0 inches for the product thickness. This calculation yielded the predicted lifetime R/inch values. Each predicted lifetime R/inch value was divided by the initial R/inch value (obtained from Table 10) to produce a ratio for each participants data set. These ratio values are given for board Set 1 and board Set 2 in Tables 12, 13, 14, and 15. Table 12 contains the ratio of lifetime R/inch for 10 years to the initial R/inch for thickness 1.5 inches using the data for Set 1 boards (seven data sets) and Set 2 boards (eight data sets) obtained by the individual participants. Similarly, Table 13 (10 years for 2 inches), Table 14 (20 years for 1.5 inches), and Table 15 (20 years for 2.0 inches).

Tables 16 and 17 summarize the results given in these four tables for Sets 1 and 2, respectively. Table 16 shows for the Set 1 boards (test boards prepared on November 29, 1993) seven data sets had one standard deviation of 1.1% for 10 year lifetimes and 1.3% for 20-year lifetimes for the ratio of predicted lifetime R/inch value to initial R/inch value for 1.5-inch thickness. Table 17 shows for the Set 2 boards (test boards prepared on January 3, 1994 that showed significant aging was occurring in the specimens prepared after November 29, 1993) the corresponding standard deviations for eight data sets were much greater (6.0% for 10 years and 6.6% for 20 years) due to the variation in initial R/inch values. These two data sets show that all test specimens of reduced thickness should be prepared at the same time in order to limit the uncertainty in predictions.

Table 16 shows the ratio increased from 0.673 to 0.688 for 10-year predictions, which is as expected for the increase in the thickness from 1.5 to 2.0 inches. Similarly, Table 16 shows the ratio increased from 0.655 to 670 for the 20 year predictions, which is as expected for the increase in thickness from 1.5 to 2.0 inches. Furthermore, the ratio values for 20-year predictions are lower than the ratio for 10-year predictions because of the longer times. Table 17 shows the same type result in the ratio values, although the standard deviations are significantly larger than those for Set 1 boards shown in Table 16.

Table 12. Ratios of predicted lifetime R/inch for 10 years to initial R/inch for thickness of 1.5 inches using the data for Set 1 and Set 2 boards obtained by the individual participants.

Set Board Numbers (Thick/Thin)	Set 1 Ratio of Predicted Lifetime R/inch for 10 years to Initial R/inch for 1.5-Inch Thickness ($h \cdot ft^2 \cdot {}^\circ F/Btu \cdot in.$)	Set 2 Ratio of Predicted Lifetime R/inch for 10 years to Initial R/inch for 1.5-Inch Thickness ($h \cdot ft^2 \cdot {}^\circ F/Btu \cdot in.$)
A. 3 / 18, 19, 20, 21	0.666	
B. 1 / 10, 11, 12, 13	0.666 L	
C. 49 / 75, 76, 77, 78		0.681 L
D. 47 / 67, 68, 69, 70		0.746
E. 6 / 30, 31, 32, 33	0.685 H	
F. 5 / 26, 27, 28, 29	0.680	
G. 2 / 14, 15, 16, 17	0.677	
H. 4 / 22, 23, 24, 25	0.668	
I. 45 / 56, 58, 59, 61		0.694
J. 48 / 71, 72, 73, 74		0.715
K. 83 / 87, 88, 91, 92		0.815 H
L. 46 / 63, 64, 65, 66		0.760
M-1. 7 / 34, 35, 36, 37	0.671	
M-2. 50 / 55, 57, 60, 80		0.737
M-3. 82 / 85, 86, 89, 90		0.700

Table 13. Ratios of predicted lifetime R/inch for 10 years to initial R/inch for thickness of 2.0 inches using the data for Set 1 and Set 2 boards obtained by the individual participants.

Set Board Numbers (Thick/Thin)	Set 1 Ratio of Predicted Lifetime R/inch for 10 years to Initial R/inch for 2.0-inch Thickness ($h \cdot ft^2 \cdot °F/Btu \cdot in.$)	Set 2 Ratio of Predicted Lifetime R/inch for 10 years to Initial R/inch for 2.0-inch Thickness ($h \cdot ft^2 \cdot °F/Btu \cdot in.$)
A. 3 / 18, 19, 20, 21	0.680	
B. 1 / 10, 11, 12, 13	0.683	
C. 49 / 75, 76, 77, 78		0.706 L
D. 47 / 67, 68, 69, 70		0.758
E. 6 / 30, 31, 32, 33	0.702 H	
F. 5 / 26, 27, 28, 29	0.695	
G. 2 / 14, 15, 16, 17	0.689	
H. 4 / 22, 23, 24, 25	0.680 L	
I. 45 / 56, 58, 59, 61		0.709
J. 48 / 71, 72, 73, 74		0.730
K. 83 / 87, 88, 91, 92		0.826 H
L. 46 / 63, 64, 65, 66		0.780
M-1. 7 / 34, 35, 36, 37	0.689	
M-2. 50 / 55, 57, 60, 80		0.749
M-3. 82 / 85, 86, 89, 90		0.714

Table 14. Ratios of predicted lifetime R/inch for 20 years to initial R/inch for thickness of 1.5 inches using the data for Set 1 and Set 2 boards obtained by the individual participants.

Set Board Numbers (Thick/Thin)	Set 1 Ratio of Predicted Lifetime R/Inch for 20 years to Initial R/inch for 1.5-Inch Thickness ($h{\cdot}ft^2{\cdot}°F/Btu{\cdot}in.$)	Set 2 Ratio of Predicted Lifetime R/Inch for 20 years to Initial R/inch for 1.5-Inch Thickness ($h{\cdot}ft^2{\cdot}°F/Btu{\cdot}in.$)
A. 3 / 18, 19, 20, 21	0.650	
B. 1 / 10, 11, 12, 13	0.645 L	
C. 49 / 75, 76, 77, 78		0.645 L
D. 47 / 67, 68, 69, 70		0.732
E. 6 / 30, 31, 32, 33	0.664	
F. 5 / 26, 27, 28, 29	0.659	
G. 2 / 14, 15, 16, 17	0.667 H	
H. 4 / 22, 23, 24, 25	0.654	
I. 45 / 56, 58, 59, 61		0.676
J. 48 / 71, 72, 73, 74		0.698
K. 83 / 87, 88, 91, 92		0.800 H
L. 46 / 63, 64, 65, 66		0.731
M-1. 7 / 34, 35, 36, 37	0.647	
M-2. 50 / 55, 57, 60, 80		0.718
M-3. 82 / 85, 86, 89, 90		0.684

Table 15. Ratios of predicted lifetime R/inch for 20 years to initial R/inch for thickness of 2.0 inches using the data for Set 1 and Set 2 boards obtained by the individual participants.

Set Board Numbers (Thick/Thin)	Set 1 Ratio of Predicted Lifetime R/Inch for 20 years to Initial R/inch for 2.0-Inch Thickness ($h \cdot ft^2 \cdot °F/Btu \cdot in.$)	Set 2 Ratio of Predicted Lifetime R/Inch for 20 years to Initial R/inch for 2.0-Inch Thickness ($h \cdot ft^2 \cdot °F/Btu \cdot in.$)
A. 3 / 18, 19, 20, 21	0.664	
B. 1 / 10, 11, 12, 13	0.662 L	
C. 49 / 75, 76, 77, 78		0.674 L
D. 47 / 67, 68, 69, 70		0.744
E. 6 / 30, 31, 32, 33	0.683 H	
F. 5 / 26, 27, 28, 29	0.676	
G. 2 / 14, 15, 16, 17	0.676	
H. 4 / 22, 23, 24, 25	0.665	
I. 45 / 56, 58, 59, 61		0.692
J. 48 / 71, 72, 73, 74		0.712
K. 83 / 87, 88, 91, 92		0.812 H
L. 46 / 63, 64, 65, 66		0.756
M-1. 7 / 34, 35, 36, 37	0.667	
M-2. 50 / 55, 57, 60, 80		0.734
M-3. 82 / 85, 86, 89, 90		0.698

Table 16. Summary of ratios of predicted lifetime R/inch for 10 and 20 years for thicknesses of 1.5 and 2.0 inches obtained using the data for Set 1 boards (only) obtained by the individual participants.

A. Summary of Predictions for 10 Years

Thickness, Inch	1.5	2.0
Average Ratio	0.673	0.688
High Ratio	0.685	0.702
Low Ratio	0.666	0.680
Range, Ratio	0.019 2.82%	0.022 3.20%
One Standard Deviation	1.11%	1.19%

B. Summary of Predictions for 20 Years

Thickness, Inch	1.5	2.0
Average Ratio	0.655	0.670
High Ratio	0.667	0.683
Low Ratio	0.645	0.662
Range, Ratio	0.022 3.36%	0.021 3.13%
One Standard Deviation	1.29%	1.18%

Table 17. Summary of ratios of predicted lifetime R/inch for 10 and 20 years for thicknesses of 1.5 and 2.0 inches obtained using the data for Set 2 boards (only) obtained by the individual participants.

A. Summary of Predictions for 10 years

Thickness, Inch	1.5	2.0
Average Ratio	0.731	0.746
High Ratio	0.815	0.826
Low Ratio	0.681	0.706
Range, Ratio	0.134 18.3%	0.120 16.1%
One Standard Deviation	5.95%	5.54%

B. Summary of Predictions for 20 years

Thickness, Inch	1.5	2.0
Average Ratio	0.711	0.728
High Ratio	0.800	0.812
Low Ratio	0.645	0.674
Range, Ratio	0.155 21.8%	0.138 19.0%
One Standard Deviation	6.58%	6.03%

CONCLUSIONS

The results obtained from 13 participating laboratories proved this interlaboratory comparison was a successful demonstration of the PIMA Standard to estimate the long-term thermal resistance of unfaced, rigid, closed-cell, polyisocyanurate foam insulation as a function of time using specimens of reduced thickness. The material for this demonstration was an experimental polyisocyanurate boardstock blown with HCFC-141b and CO_2. The test results were obtained on thick and thin specimens using heat flow meter apparatuses that are capable of measuring apparent thermal conductivity in accordance with ASTM C 518 Test Method.

A computer program was developed and used to predict the lifetime thermal resistance (R/inch) of foam insulations.

The ratios of predicted lifetime R/inch value to initial R/inch value for the seven data sets on board Set 1 had one standard deviation of 1.1% for 10-year lifetimes and 1.3% for 20-year lifetimes for a thickness of 1.5 inches.

The ratios of predicted lifetime R/inch value to initial R/inch value for the eight data sets on board Set 2 had one standard deviation of 6.0% for 10-year lifetimes and 6.6% for 20-year lifetimes for a thickness of 1.5 inches. This clearly shows that all test specimens of reduced thickness should be prepared at the same time.

The results of this study provide a needed precision and bias statement for the PIMA Standard, Estimating the Long-Term Thermal Performance of Unfaced, Rigid, Closed-Cell, Polyisocyanurate (PIR) Foam Insulation, and for the ASTM C16 Standard, Estimating the Long-Term Change in the Thermal Resistance of Unfaced Rigid Closed Cell Plastic Foams by Slicing and Scaling Under Controlled Laboratory Conditions.

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REFERENCES

1. ASTM C 1114-92, " Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Thin-Heater Apparatus", pp. 598-606, Volume 04.06, 1994 Annual Book of ASTM Standards, 1994.

2. ASTM C 518-91, "Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus", pp. 150-161, Vol. 04.06, 1994 Annual Book of ASTM Standards, 1994.

3. McElroy, D. L., Graves, R. S., Yarbrough, D. W., and Weaver, F. J.,Thermal Resistance of Polyisocyanurate Foam Board Insulation Blown with CFC-11 Substitutes - A Cooperative Industry/Government Project, ORNL/TM - 11645, September 1991.

4. Christian, J. E., Courville, G. E. , Desjarllais, A. O., Graves, R. S., Linkous, R. L., McElroy, D. L., Weaver, F. J., Wendt, R. L., and Yarbrough, D. W., The Technical Viability of Alternative Blowing Agents in Polyisocyanurate Roof Insulation - A Cooperative Industry/Government Project, ORNL/M-2480, December 1992.

5. Christian, J. E., Courville, G. E., Desjarlais, A. O., Graves, R. S., Linkous, R. L., McElroy, D. L., Weaver, F. J., Wendt, R. L., and Yarbrough, D. W.,The Technical Viability of Alternative Blowing Agents in Polyisocyanurate Roof Insulation: A Cooperative Industry/Government Project, ORNL/CON-367 , June 1993.

6. ASTM C X3.7 (Draft 10), "Standard Test Method for Estimating the Long-Term Change in the Thermal Resistance of Unfaced Rigid Closed Cell Plastic Foams by Slicing and Scaling Under Controlled Laboratory Conditions". This test method is under the jurisdiction of ASTM Committed C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurements. In November 1994 this document was at ASTM Society ballot and is expected to be in the 1995 Annual Book of ASTM STANDARDS.

7. Graves, R. S., McElroy, D. L., and Kollie, T. G., " Evaluation of a Commercial, Computer-Operated Heat Flow Meter Apparatus", Review of Scientific Instruments 64 (7), 1961-1970, (1993).

8. ASTM C 1132-89, "Standard Practice for Calibration of the Heat Flow Meter Apparatus", pp. 620-623 in 1994 Annual Book of ASTM Standards, Vol. 04.06 (American Society for Testing and Materials, Philadelphia, 1994).

APPENDICES

APPENDIX A

SUMMARY OF PIMA STANDARD

PIMA Standard for Estimating the Long-Term Thermal Resistance of Unfaced, Rigid, Closed-Cell, Polyisocyanurate (PIR) Foam Insulation - (PIMA Standard for Describing R(time))

This PIMA Standard uses ASTM Test Method C 518 for periodic determinations of the thermal resistance of specimens of reduced thickness for a period of 180 days to observe the effects of aging. The set of thermal resistance results for specimens aged under controlled laboratory conditions may be used to estimate the thermal performance of the foam product under actual use conditions.

The specimens are prepared from the center of a full-thickness section in order to have a cell gas content that is representative of the section at the time of manufacture. A minimum of five thermal resistance specimens per sample shall be prepared: a full thickness specimen, typically 1.5 $\pm$ 0.2 inches, that is representative of the product, and four thin specimens, 0.39 $\pm$ 0.04 inches thick. The effective specimen thickness of the thermal resistance specimen is determined to 1% using a vernier caliper or the C 518 apparatus. Each thermal resistance test is done at a mean temperature of 75 $\pm$ 4°F with a temperature difference of either 40°F or 50°F. The specimens shall be conditioned at 75 $\pm$ 4°F and 50 $\pm$ 5 percent relative humidity before and during the test sequence. At least eight data points spanning an aging period of 180 days shall be obtained for each type of specimen.

The nonlinear increase in k with time/(thickness)^2 can be described by two linear regions if one plots the natural logarithm of k versus (time)$^{1/2}$/thickness. The data are fitted using a least squares method to a straight line for region 1 and region 2. This procedure assumes the thermal conductivity (k) can be described by an exponential dependence on diffusion coefficient (D), time (t) and thickness (h).

$$Y = A + B Z \tag{1}$$

$$\ln k = \ln k_0 + (Dt)^{1/2}/h \tag{2}$$

where k_0 is the initial thermal conductivity, A is ln k_0, Y is ln k, Z is $t^{1/2}/h$, and B is $D^{1/2}$. The fitted equations for region 1 and region 2 are used to estimate k for a foam of a specific thickness at various times and the time-averaged thermal resistivity (1/k).

APPENDIX B

PIMA Standard for Describing R(time)

PIMA Standard for Estimating the Long-Term Thermal Performance of Unfaced, Rigid, Closed-Cell, Polyisocyanurate (PIR) Foam Insulation (PIMA Standard for Describing R(time))

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Inch-pound units are primary; Metric or S. I. units are secondary.

PIMA Standard for Estimating the Long-Term Thermal Resistance of Unfaced, Rigid, Closed-Cell, Polyisocyanurate (PIR) Foam Insulation - (PIMA Standard for Describing R(time))

1. SCOPE

   1.1 This PIMA Standard uses ASTM Test Method C 518 for periodic determinations of the thermal resistivity of specimens of reduced thickness which shortens the time required to observe the effects of aging.
   1.2 The set of thermal resistivity results for specimens of an unfaced, rigid, closed-cell, PIR foam aged for 180 days under controlled laboratory conditions may be used to provide an acceptable description of the thermal performance of the unfaced foam product under actual use conditions.
   1.3 Annexes provide detailed descriptions of procedures to make the measurement and to interpret the results.

2. APPARATUS

   2.1 The thermal resistivity test apparatus shall conform to the latest version of ASTM Test Method C 518.
   2.2 The apparatus shall be capable of testing one single full-thickness specimen and a stack of thin specimens with a total thickness of less than 1.6 inches (40 mm) with an estimated error in thickness of not more than 2 % attributed to errors in plate and specimen flatness and parallelism (0.032 inches, 0.8 mm).
   2.3 The apparatus must be capable of testing at a mean temperature of 75 ± 4°F (24 ± 2°C) with a temperature difference of either 40°F (22°C) or 50°F (28°C).
   2.4 The humidity within the test apparatus shall be maintained low enough to prevent moisture condensation on the cold plate(s) of the apparatus. For optimum results, the measuring apparatus should be used under normal conditions, 75 ± 4°F (24 ± 2°C) and 50 ± 5% relative humidity.

3. SAMPLING

   3.1 The data analyses used in this PIMA Standard assumes that the reduced-thickness specimen has the same diffusion coefficients and initial cell gas composition as the material. The sampling process and multiple tests on specimens of two thicknesses can help assure that these conditions are met.
   3.2 Specimens shall be prepared as soon as possible after production so that the cell gas content within the sample is representative of initial conditions. Specimens prepared from the center of a full thickness section may still have a cell gas content that is representative of the section at the time of manufacture.
   3.3 (ADDED AFTER COMPLETING THE 1994 INTERLABORATORY COMPARISON M-REPORT) All test specimens of reduced thickness should be prepared at the same time to limit uncertainty in predictions.

4. THERMAL RESISTIVITY SPECIMENS

4.1 This PIMA Standard uses two types of specimens with flat surfaces: a full-thickness specimen typically 1.5 ± 0.3 inches (38 ± 8 mm) that is representative of the product, and four thin specimens 0.39 ± 0.1 inches (10 ± 3 mm) thick. Thus, a minimum of five thermal resistivity specimens per sample shall be prepared to allow thermal tests on stacked thin specimens of nominal thickness 1.2 to 2.0 inches (30 to 51 mm).

4.2 The full-thickness specimen is formed from a full-thickness section whose cross-sectional area is large enough to fully cover the plate area of the test apparatus. The full-thickness specimen and the thin specimens are formed by reducing the thickness of this section by alternatively removing thin layers from each major surface.

5. MEASUREMENT OF SPECIMEN THICKNESS

5.1 The effective specimen thickness of each thermal resistivity specimen is determined by either (1) 12 measurements of the thickness around the full-thickness specimen perimeter with a dial-gage or vernier caliper to ±0.001 inch, or (2) direct thickness measurement of the specimen when it is in the C 518 apparatus and after the apparatus has been calibrated for thickness. As shown in the Annex, (See Table A5), the latter thickness may be less than the former thickness by as much as 1 percent, because of compression effects.

6. THERMAL RESISTIVITY MEASUREMENT

6.1 Weigh each thermal resistivity specimen to within 10 mg. Measure the cross-sectional area of the specimen to within 0.01 inches (0.2 mm). Use this area and the effective specimen thickness (See 5.1) to compute the specimen volume and test density.

6.2 Conduct the thermal resistivity test at a mean temperature of 75 ± 4°F (24 ± 2°C) with a temperature difference of either 40°F (22°C) or 50°F (28°C) imposed on the specimen.

6.3 For a series of tests on a specimen as a function of time, the specimens shall be conditioned at 75 ± 4°F (24 ± 2°C) and 50 ± 5 percent relative humidity before and during the test sequence for this PIMA Standard.

6.4 The major flat surfaces of the specimens shall not be in physical contact during horizontal or vertical storage for aging.

6.5 Because of the thinness of the aging specimens, this PIMA Standard uses four identical layers stacked together to fabricate a thick thermal resistivity specimen. The stacked specimen must be oriented the same for each test. The specimen should be loaded and tested in the apparatus specimen chamber with the same orientation for each test time. This PIMA Standard requires at least nine data points spanning an aging period of 180 days for each type of specimen. Specific foams may age at different rates. The test times should be selected to yield data for both of the two aging processes. A two week/four week test cycle that spans aging for 180 days after specimen preparation is: Test 1: 14 days (time after sample preparation); Test 2: 28 days; Test 3: 42 days; Test 4: 56 days; Test 5: 84 days; Test 6: 112 days; Test 8: 168 days; Test 9: 196 days. This test cycle allows tests to be conducted at the same time each week, for example, each Monday morning.

7. REPORT

The report shall give the following information, including references to applicable test methods. This information may be organized as indicated on the fill-in-the-blanks Laboratory Report Form as shown here and in the Annex. The data summary tables are the basis for describing the performance of products as a function of time under actual use conditions.

7.1 The name, address, and any other identification of the test laboratory and the date of the report.
7.2 The name and any other identification of the material tested, and the date of the test.
7.3 The manufacturer of the material, the date obtained, the date of manufacture and/or the manufacturers lot number.
7.4 The method of specimen preparation.
7.5 The specimen dimensions and weight, and the calculated test density.
7.6 The aging conditions.
7.7 The type, size, and meter area of the thermal test apparatus.
7.8 The test mean temperature, test temperature difference, and age of the specimen at each test time.
7.9 The specimen apparent thermal conductivity and thermal resistivity at each test time.
7.10 If applicable, a plot of the thermal resistivity as a function of time and a table of the time-averaged thermal resistivity for several times selected as service life.
7.11 A statement of the precision and bias of the test apparatus and procedure used to arrive at the reported data.(See ASTM C 518)
7.12 Any additional pertinent observations and remarks.

TEST LABORATORY REPORT FORM DATE____________

7.1 Test Laboratory Information Name Street Address City, State, Zip	
7.2 Material Tested Date of Test Name of Person doing the tests	
7.3 Manufacturer of Material Material Lot Number Date of Production Date Obtained	
7.4 Method of Specimen Preparation Planer; Band Saw; Slicer; Other.	
7.5 Specimen Characteristics Thickness in Inches Length in Inches; Width in Inches. Specimen Weight in Pounds (grams) Specimen Density in Pounds per Cubic Foot	
7.6 Specimen Aging Conditions 75 °F; Other	
7.7 Thermal Test Apparatus Type R-Matic; K-Matic; Anacon (may not be allowed for tests run by C 518); Other.	
7.8 Test Conditions Mean Test Temperature: 75°F; Other. Temperature Difference: 50°F; 40°F; Other. Specimen Age in Days when Tested.	
7.9 Specimen Properties Obtained Apparent Thermal Conductivity in Btu*in/h*ft^2*°F Thermal Resistivity in (h*ft^2°F)/Btu*in)	

DATA UTILIZATION AND CALCULATED PROPERTIES FORM

1. SUMMARY OF TEST DATA

TEST DATE	TEST NUMBER	SPECIMEN AGE (d) and THICKNESS (h) WHEN TESTED	APPARENT THERMAL CONDUCTIVITY (k)	NATURAL LOG OF 100 * k

2. CALCULATED AGED SPECIMEN CHARACTERISTICS.

TEST NUMBER	t/h^2 in d/in^2	$(t/h^2)^{1/2}$ in $(d/in^2)^{1/2}$

3. SUMMARY OF DATA FIT BY LEAST-SQUARES METHOD

REGION	A	B	NUMBER OF DATA POINTS	INTERCEPT

4. CALCULATED PROPERTIES

PERIOD OF TIME IN YEARS	TIME-AVERAGED THERMAL RESISTIVITY
1	
2	
5	
10	

TIME-AVERAGED THERMAL RESISTIVITY____________________
THERMAL RESISTIVITY AT 5 YEARS______________________

8. REFERENCES

1) McElroy, D. L., Graves, R. S., Yarbrough, D. W. and Weaver, F. J., Laboratory Test Results on the Thermal Resistance of Polyisocyanurate Foamboard Insulation Blown with CFC-11 Substitutes - A Cooperative Industry/Government Project, ORNL/TM-11645, September 1991.

2) Christian, J. E., Courville, G. E., Graves, R. S., Linkous, R. L., McElroy, D. L., Weaver, F. J., and Yarbrough, D. W., Thermal Measurement of In-Situ and Thin-Specimen Aging of Experimental Polyisocyanurate Roof Insulation Foamed with Alternative Blowing Agents, Insulation Materials, Testing and Applications, 2nd Volume, ASTM STP 1116, R. S. Graves and D. C. Wysocki, Eds. American Society for Testing and Materials, Philadelphia, 1991, pp. 142-166.

3) Document CX3.7 (Draft 6) January, 1992. Standard Test Method for Estimating the Long Term Change in the thermal resistance of Unfaced Rigid Closed Cell Plastic Foams by Slicing and Scaling. Under consideration by ASTM Committee C16 on Thermal Insulation.

4) Bomberg, M. T., Scaling Factors in Aging of Gas-filled Cellular Plastics, Journal of Thermal Insulation, Volume 13, January, 1990, p. 149.

5) Edgecombe, F. H., Progress in Evaluating Long-Term Thermal Resistance of Cellular Plastics, CFCS & Polyurethane Industry: Volume 2. A Compilation of Technical Publications 1988-1989. F. W. Lichtenburg, Ed., Technomic Publishing Co., pp. 17-24.

6) Norton, F. J., Thermal Conductivity and Life Polymer Foams, Journal of Cellular Plastics, 1967, pp. 23-37.

7) Ball, J. S., Healey, G. W., and Partington, J. B., Thermal Conductivity of Isocyanate-Based Rigid Cellular Plastics: Performance in Practice, European Journal of Cellular Plastics, 1978, pp. 50-62.

8) Mullenkamp, S. P., and Johnson, S. E., In-Place Thermal Aging of Polyurethane Foam Roof Insulations, 7th Conference on Roofing Technology, National Roofing Contractors Association, 1983.

9) Booth, R. J., R-Value Aging of Rigid Urethane Foam Products, Proceedings, Society of Plastics Industry of Canada, 1980.

10) McElroy, D. L., Graves, R. S., Weaver, F. J., and Yarbrough, D. W., The Technical Viability of Alternative Blowing Agents in Polyisocyanurate Roof Insulation, Part 3: Acceleration of Thermal Resistance Aging Using Thin Boards, Polyurethanes 90 Conference Proceedings, Orlando, FL, 1991.

11) Scheutz, M. A., and Glicksman, L. R., A Basic Study of Heat Transfer Through Foam Insulations, Proceedings of the Sixth International Polyurethane Conference, San Diego, CA, 1983, pp. 341-347.

12) Ostrogorsky, A. G., Aging of Polyurethane Foams, D.Sc. thesis at MIT, L. R. Glicksman, Supervisor, Cambridge, MA, 1985.

13) Schwartz, N. V., Bomberg, M. T., and Kumaran, M. K., Measurements of the Rate of Gas Diffusion in Rigid Cellular Plastics, Journal of Thermal Insulation, Volume 13, 1989, pp. 48-61.

14) Normandin, N. and Kumaran, M. K., A Pressure-Volume Apparatus to Measure the Effective Thickness of Cellular Plastic Test Specimens, Journal of Thermal Insulation, Volume 15, 1992.

15) Graves, R. S., McElroy, D. L., Miller, R. G., Yarbrough, D. W., and Zarr, R. R., Interlaboratory Comparison of Four Heat Flow Meter Apparatuses on Planed Polyisocyanurate Boards Foamed with CFC-11, Oak Ridge National Laboratory Report ORNL/TM-11720, January, 1991.

REFERENCES IN *Annual Book of ASTM Standards*, Vol 04.06.

C 168 Definitions of Terms Relating to Thermal Insulating Materials

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of a Guarded Hot Plate Apparatus

C 236 Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box

C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of The Heat Flow Meter Apparatus

C 578 Specification for Preformed, Cellular Polystyrene Thermal Insulation

C 591 Specification for Unfaced Preformed Rigid Cellular Polyurethane Thermal Insulation

C 976 Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Calibrated Hot Box

C 984 Specification for Perlite Board, Rigid Cellular Polyurethane Composite Roof Insulation

C 1013 Specification for Membrane Faced Rigid Cellular Polyurethane Roof Insulation

C 1029 Specification for Spray-Applied Rigid Cellular Polyurethane Roof Insulation

C 1045 Practice for Calculating Thermal Transmission Properties From Steady—State Heat Flux Measurements

C 1050 Specification for Rigid Cellular Polystyrene-Cellulosic Fiber Composite Roof Insulation

C 1114 Test Method for Steady-State Thermal Transmission Properties by Means of The Thin Heater Apparatus

C 1126 Specification for Faced or Unfaced Rigid Cellular Phenolic Thermal Insulation

9. TERMINOLOGY AND SYMBOLS

1. Definitions – For definitions of terms and symbols used in this proposal, refer to Definitions C 168.

2. Description of Terms Specific to this PIMA Standard

2.1 aging -- the change in thermophysical properties of rigid closed-cell PIR foam with time due to changes in the composition of the gas contained within the cells.

2.2 effective diffusion coefficient -- a material property which relates the rate of gas transport to the gas pressure difference across the material of a given thickness at a given temperature.

2.3 specimen thickness -- the actual thickness as determined by tests using a plane table.

2.4 primary stage – that portion of the aging process where changes in thermophysical properties are primarily influenced by the diffusion of air components into the rigid closed-cell PIR foam.

2.5 scaling factor – the ratio of the square of the material thickness and the square of the test specimen thickness. This ratio represents the acceleration rate which is being applied to the aging process of a rigid closed cell plastic foam because of thickness differences.

2.6 secondary stage – that portion of the aging process where changes in thermophysical properties are primarily influenced by the diffusion of blowing agent(s) from the rigid closed cell PIR foam.

2.7 service life -- the anticipated period of time that the material is expected to maintain the manufacturers claimed thermophysical properties. The service life may be dependent on the specific end-use application.

2.8 time-averaged thermal resistance -- the thermal resistance of a material of given thickness averaged over a specified time period.

2.9 transfer point (T_t) -- the estimated age of a rigid closed cell plastic foam when the aging process switches from the primary to secondary stage (see Figures 1 and 2).

3.3 Symbols

B	=	constant,
D	=	diffusion coefficient, cm^2/sec,
$\Delta X1$	=	specimen (material) thickness, inches (m),
ΔX_2	=	specimen thickness, inches (m),
k	=	apparent thermal conductivity, $Btu \cdot in/(h \cdot ft^2 \cdot °F)$ $(W/(m \cdot K))$,
k_g	=	thermal conductivity of the cell gas mixture, $Btu \cdot in/(h \cdot ft^2 \cdot °F)$ $(W/(m \cdot K))$,
k_o	=	initial apparent thermal conductivity, $Btu \cdot in/(h \cdot ft^2 \cdot °F)$ $(W/(m \cdot K))$,
k_r	=	thermal conductivity due to thermal radiation, $Btu \cdot in/(h \cdot ft^2 \cdot °F)$ $(W/(m \cdot K))$,
k_s	=	thermal conductivity of the solid polymer, $Btu \cdot in/(h \cdot ft^2 \cdot °F)$ $(W/(m \cdot K))$,
p	=	partial pressure, atm,
r	=	thermal resistivity, $(h \cdot ft^2 \cdot °F)/Btu \cdot in.$ $((m \cdot K)/W)$
R	=	thermal resistance, $(h \cdot ft^2 \cdot °F)/Btu$ $((m^2 \cdot K)/W)$,
R_o	=	initial thermal resistance, $(h \cdot ft^2 \cdot °F)/Btu$ $((m^2 \cdot K)/W)$,
R_a	=	estimated time-averaged resistance, $(h \cdot ft^2 \cdot °F)/Btu$ $((m^2 \cdot K)/W)$,
R_t	=	thermal resistance on t^{th} day, $(h \cdot ft^2 \cdot °F)/Btu$ $((m^2 \cdot K)/W)$,
t	=	time, days, d,
t_a	=	age of specimen when the instantaneous thermal resistance is equal to a time-averaged thermal resistance, days, d,
t_m	=	service life, days, d,
t_t	=	transfer point, days, d,
h	=	specimen test thickness, inches (m).

10. ANNEXES

Annex A. The Aging Process

A.1 - Rigid closed cell plastic foam insulations are produced by foaming various plastic polymers. At the time of manufacture, the cells of a PIR foam usually contain their highest percentage of blowing agent and the lowest percentage of air components. As time passes, the relative concentrations of these gases change due to diffusion, resulting in a decrease in the thermal resistivity due to an increase in the thermal conductivity of the resultant cell gas mixture. This assumes the blowing agent(s) is a gas whose apparent thermal conductivity is less than that of air and effective diffusion coefficient are lower than those of the air components. This assumption is made so that the text is clear; if the blowing agent diffuses faster than the air components, definitions of the stages of aging would require modification and any discussions regarding diffusion rates would need to be changed.

A.2 - The loss in thermal resistivity due to the phenomena described in Section A.1 occurs over an extended period of time. Information regarding changes in the thermal resistivity of these materials is required so that rational decisions regarding formulations, production, and comparisons with other materials can be made. Ideally, time—averaged thermal resistance data for the expected service life should be available after as short a period as possible.

A.3 - ASTM Standards C 578, C 591, C 984, C 1013, C 1029, C 1050, and C 1126 on rigid closed cell plastic foams indicate this decrease in thermal resistivity occurs over an extended period of time. However, these standards currently require that freshly manufactured foams be measured for thermal resistivity after conditioning at 73 +/- 2°F (23 +/- 1°C) for 180 +/- 5 days from the time of manufacture, or at 140 +/- 2°F (60 +/- 1°C) for 90 days. These standards do not currently specify long-term or time-averaged thermal resistivity.

A.4 - This procedure requires that the material characteristics of the thin specimens are equivalent to those of the material under investigation. In particular, the specimens of reduced thickness must have the same effective diffusion coefficient and initial cell gas content as those of the full thickness material, and that one-dimensional diffusion dominates, limiting the application of this test procedure to unfaced or faced-with-a-permeable-material "homogeneous" materials.

A.5 - The procedure outlined in this method can be used to produce a characteristic aging curve (relationship between the thermophysical properties with time). This relationship has been used by researchers to calculate effective diffusion coefficients. (1, 2).

A.6 - During the service life of a rigid closed cell PIR foam, air components diffuse into the cells, and the blowing agent diffuses out of the cells or partially dissolves in the solid polymer matrix. Each process occurs at a rate that depends on the type of polymer, the foam structure, the temperature, the gas type and its pressure.

A.7 - Since the inward diffusion of air components is generally much faster than the outward diffusion of the captive blowing agent, the aging process proceeds in two stages. During the first stage, the cell gas composition changes at a significant rate because of the rapid diffusion of air components into the cell and the outward diffusion of all rapidly diffusing blowing agents, if present. These compositional changes cause the thermal resistivity of the material to change. This stage of aging is defined as the primary stage.

A.7.1 - If carbon dioxide is used as a blowing agent or is generated during foam manufacture, its outward diffusion rate will usually exceed the entry rate of air components during the primary stage.

A.7.2 - As the diffusion of air components nears completion, the thermal resistivity of the material changes more slowly. The thermal resistivity continues to change, however, due to continuing outward diffusion from the cells of the blowing agent. This stage is defined as the secondary stage.

A.7.3 - A number of researchers studying aging have depicted their thermal performance data (thermal conductivity, thermal resistance, thermal resistivity) for unfaced, rigid, closed-cell plastic foams as a function of the logarithm of time. See Refs (4), (5), (6), (7), (8), and (9).

$$R = F\{\ln t\} \tag{A1}$$

Figures 1 and 2 are examples of aging curves for an unfaced, rigid, closed-cell plastic foam utilizing this technique (3). The inflection between the two stages of the aging process is defined as the transfer point. A second transition may occur, depending on the blowing agent(s) used.

A.7.4 - The apparent thermal conductivity of a rigid closed-cell plastic foam, k, can be approximately expressed as the sum of the apparent thermal conductivities due to radiation (k_r), due to the gas mixture (k_g), and due to the solid polymer (k_s). See Refs. (1,11) for further details.

$$k = k_r + k_g + k_s \tag{A2}$$

A.7.5 - It is assumed that the values of k_r and k_s do not change significantly during the time the gas content within the cells is changing. Then Equation A2 implies that the aging process can be studied by exclusively investigating the change in the gas thermal conductivity, and k_g can be determined by studying the change in partial pressure of the cell gas components as a function of time. The governing parameters controlling the change in the partial pressure, p, of the gas components are their effective diffusion coefficients D, the thickness ΔX, and time t. See Ref (1). Solubility changes are not addressed. In order to accelerate the aging process, either the diffusion coefficients can be increased or the thickness reduced.

A.7.6 - Diffusion coefficients can be increased by raising the temperature (1, 12). The amount of acceleration achievable by raising the aging temperature depends on the temperature dependence of the diffusion coefficients. A specific increase in temperature does not equally change the diffusion coefficients of all the gases involved in the aging process. A possible third limitation is that elevating the temperature can damage the cellular structure of the foam, which is suggested in Ref (13).

A.7.7 - Reducing specimen thickness can yield large increases in aging rates and does not expose the material to potentially damaging elevated temperatures. For a material satisfying the requirements of constant D and initial p, the same value of the ratio $t/\Delta X^2$ will yield the same value of p, and therefore the same values of λ_g and λ. Therefore, the thermal resistivity of a specimen of thickness ΔX_1 at time t_1 can be determined after conditioning a specimen of effective thickness ΔX_2 over a time interval t_2. The time interval t_2 can be calculated by

$$t_2 = t_1 * (\Delta X_2 / \Delta X_1)^2 \quad \text{(A3)}$$

The ratio $(\Delta X_2 / \Delta X_1)^2$ is defined as the scaling factor and Annex B shows an example calculation.(3)

A.7.8 The nonlinear increase in k with time/(thickness)^2 can be described by two linear regions if one plots the natural logarithm of k versus (time)$^{1/2}$/thickness. This is the ORNL procedure (1, 2, 10). If one assumes that k can be described by an exponential dependence on diffusion coefficient (D), time (t) and thickness (h):

$$k = k_0 \exp \{(Dt)^{1/2}/h\}, \quad \text{(A4)}$$

where k_0 is the initial thermal conductivity, then one observes the following dependence of k on t in the primary and secondary regions:

$$\ln k = \ln k_0 + (Dt)^{1/2}/h \quad \text{(A5)}$$

$$Y = A + B Z \quad \text{(A6)}$$

where $A = \ln k_0$
$Y = \ln k$
$Z = t^{1/2}/h$, and
$B = D^{1/2}$.

Thus, if one measures the k of a PIR foam product of thickness (h) as a function of aging time (t), then a plot of Y versus Z should yield a straight line of slope B. A least-squares fitting of the data to the straight line represented by Eq. A6 yields an intercept of $\ln k_0$ and a slope equal to $D^{1/2}$. Figure 3 shows this increase in k (plotted as ln 100 k for convenience) for specimens of three thicknesses of foam blown with CFC-11 and aged at 75°F. The two linear regions correspond to the primary and secondary stages. Figure 3 shows calculated and experimental data (1) for these two regions.

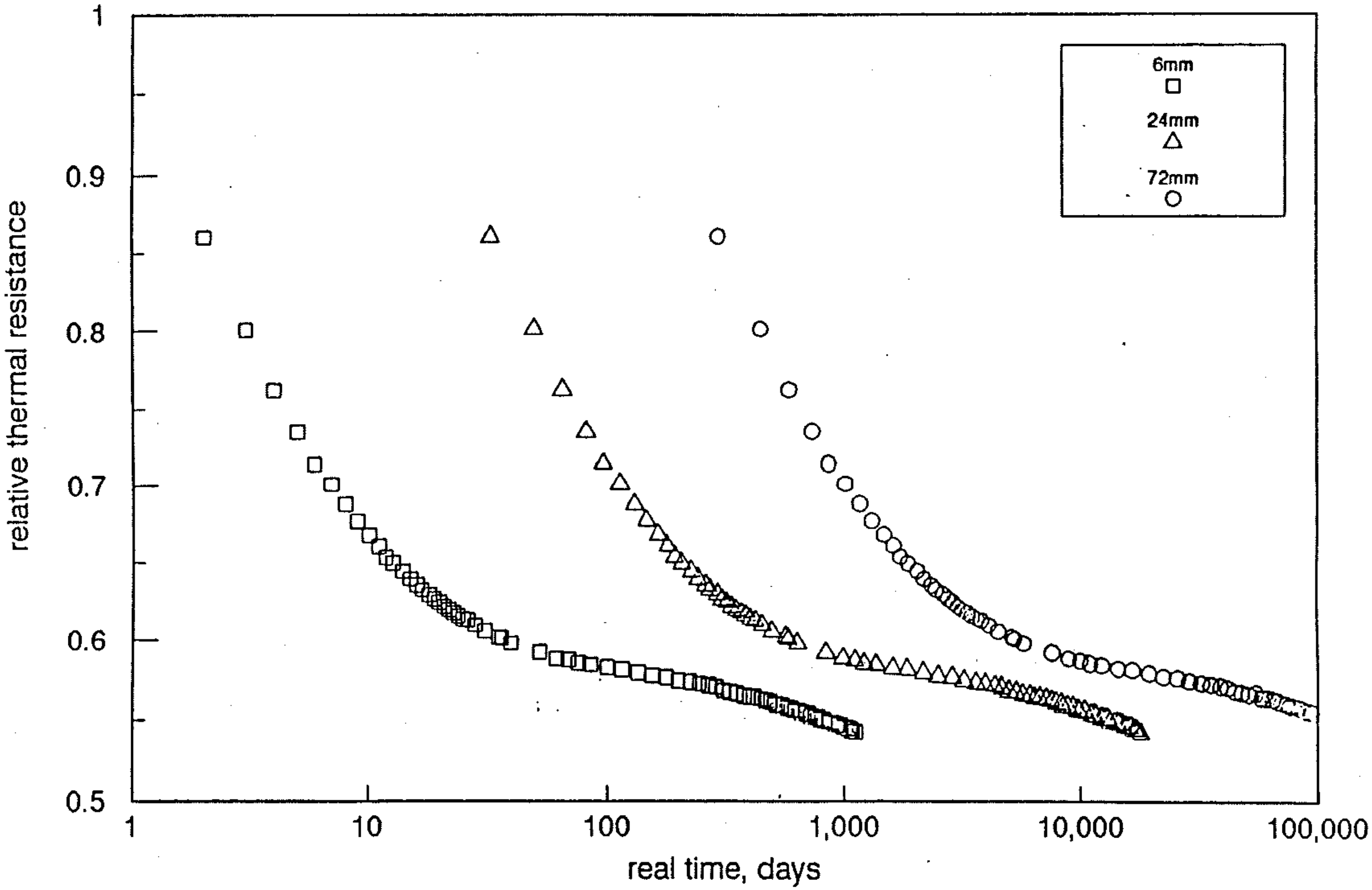


Figure 1. The relative thermal resistance of three thicknesses of a cellular plastic foam as a function of time.

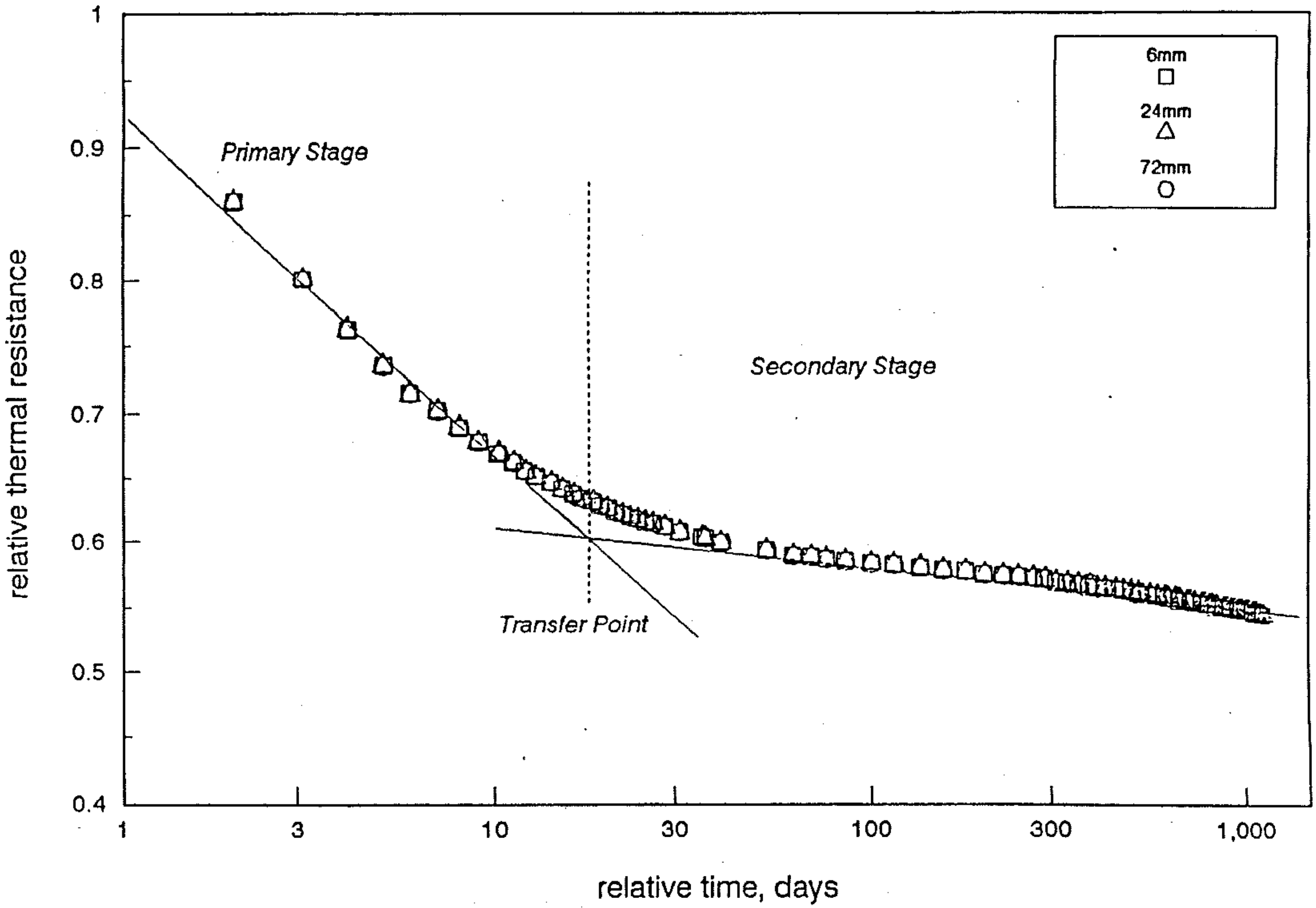


Figure 2. The relative thermal resistance of three thicknesses of a cellular plastic foam after application of the scaling factor.

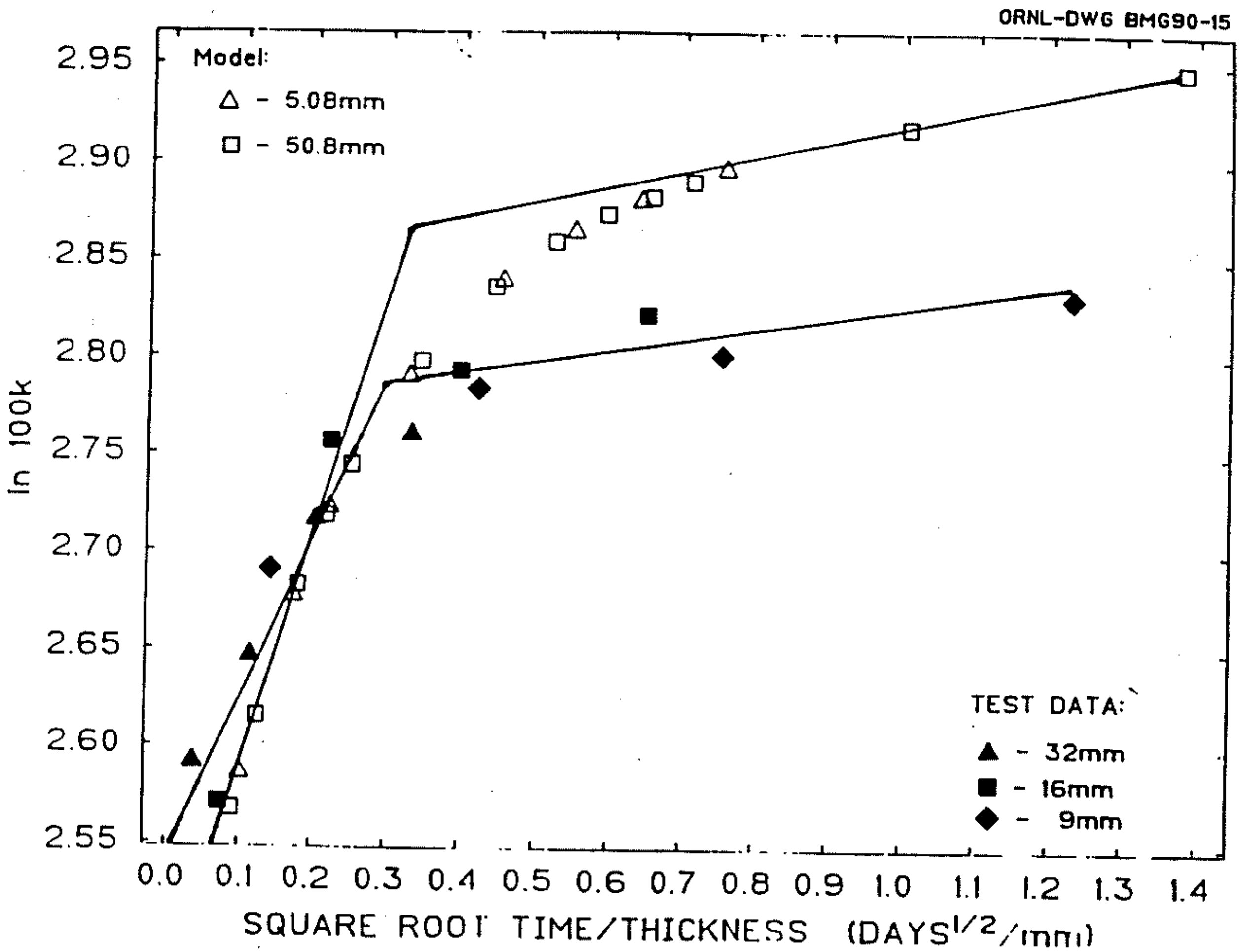


Figure 3. Test data (filled points) show the increase in k (75°F) for thin specimens of rigid board foamed with CFC-11 aging at 75°F. Massachusetts Institute of Technology model predictions are shown as unfilled points.

Annex B. Example Calculation

B.1 Determination of Scaling Factor

B.1.1 This calculation follows the procedure in Section A.

B.1.2 *Problem* -- A 2 inch (50 mm) thick "homogeneous" rigid, unfaced, closed-cell plastic foam material is to be measured for its long term thermal resistivity. An estimate of the thermal resistivity at 3650 days (10 years) is needed.

B.1.3 *Solution* -- A 0.43 inch (11 mm) thick slice is removed from the center of a section of the insulation material following the guidelines described in Annex E.

B.1.4 Using Equation A3, the specimen must be conditioned for 3650 days * $(0.43\ in)^2/(2\ in)^2$ or 169 days to obtain the equivalent thermal performance of the full thickness section aged for 3650 days (10 years).

B.1.6 The scaling factor is $(0.43/2)^2 = 0.046$.

Annex C. Utilization of Thermal Resistivity Data

C.1 *Calculation of Time-averaged Thermal Resistivity* -- Assuming that R_t is the thermal resistance of a rigid closed cell plastic foam on the t^{th} day, R_a is the time-averaged thermal resistivity, and t_m is the intended service life of the rigid closed cell plastic foam in days, then

$$R_a = 1/t_m \int_{t1}^{t_m} R_t\, dt \qquad \text{(A7)}$$

where t1 is much less than tm.

C 1.1 Assuming a logarithmic dependence of thermal resistivity with time, then the initial portion of the aging process can be approximated by

$$R_t = R_o - B\ \ln(t) \qquad \text{(A8)}$$

where R_o is the thermal resistivity of the cellular plastic on the first day of the aging period and B is a constant. Equation A8 can be expressed as

$$(R_t-R_m)/(R_o-R_m) = 1 - \ln(t)/\ln(t_m) \qquad \text{(A9)}$$

where R_m is the thermal resistivity on the last day of the aging period. Therefore R_a is equal to the thermal resistivity on the a^{th} day where

$$t_a = t_m / (2.72) \qquad \text{(A10)}$$

Annex D. Estimation of the Time-Averaged Thermal Resistivity

D.1 This calculation follows the procedure in Annex A.

D.2 *Problem* -- The same material described in Annex A has a service life of 3650 days (10 years). An estimate of the time-averaged thermal resistivity is needed.

D.3 *Solution* -- Repeat the process described in Annex B to prepare a test specimen and determine the effective thickness of 0.43 inches (11 mm).

D.4 Using Equation A10, calculate the length of conditioning that is required to obtain the equivalent to the average thermal resistivity. The average thermal resistivity or time-averaged thermal resistivity is equal to the thermal resistivity measured after 3650 days/(2.72) or about 1342 days.

D.5 Using Equation A3, the thin specimen must be conditioned for 1342 days * $(0.43\ in)^2/(2\ in)^2$ or 62 days to obtain an instantaneous thermal resistivity that is equivalent to an estimate of the time-averaged thermal resistivity for a service life of 3650 days (10 years).

Annex E. Thin-Specimen Preparation Guidelines

E.1 Specimen preparation equipment that has successfully been used to prepare thin specimens is listed below. Ref (5) summarizes these techniques and compares their effectiveness. This listing is not intended to preclude the development or use of any other demonstrated methods of specimen preparation.

E.1.1 Surface grinder or planer. This method has been used at ORNL.

E.1.2 High speed band-saw with a fine tooth blade or razor blade.

E.1.3 A combination lathe/motor-driven meat slicer.

E.2 *Surface Damage* -- Equipment for preparing thin specimens shall be selected based on the equipment's ability to reproduce the amount of surface damage (open cells) created in the preparation process. Small variations in surface damage may yield significant variability in the data obtained from the thin specimen. Different specimen preparation equipment may be required to prepare thin specimens from generically different rigid closed cell plastic foams.

E.3 *Thickness Uniformity* -- The equipment used to prepare specimens shall be capable of producing specimens whose uniformity of thickness imparts an uncertainty no greater than 2 percent to the thickness measurement.

Annex F. Precision and Bias (Deviation)

F.1 The precision of this method is significantly influenced by the specimen preparation techniques and the dimension measurement procedures as well as the precision of the thermal test method used. Precision data on the combined procedures are not yet available.

F.2 Bias (deviation) between the thin specimen aged R-Values and the long-term full thickness aged R-Values can only be determined after the actual aging of the full thickness material is completed.

F.3 An interlaboratory comparison of four laboratories performing experiments at 24°C (75°F) on three test specimens of polyisocyanurate foam ranging in thickness from 32 to 64 mm (1.3 to 2.6 inches) reported an average deviation of 0.8 percent and a two standard deviation (2σ) value of 2.3 percent. See Ref (15) for further details of this comparison. The thicknesses of the specimens in this round-robin are near those proposed by this test procedure.

F.4 The precision and bias (deviation) of the thermal resistivity projections and estimations of the time-averaged thermal resistance are presently unknown and will be a subject for a future revision of the test method.

F.5 (ADDED AFTER COMPLETING THE 1994 INTERLABORATORY COMPARISON M-REPORT) Th predicted lifetime R/inch values for the 15 data sets had one standard deviation of 1.4% for 10 year lifetimes and 1.7% for 20 year lifetimes.

Annex G. Key Words

G.1 This test method is indexed under the following terms: aging, rigid closed cell PIR foams, thermal insulation, thermal resistivity, design thermal resistivity, time-averaged thermal resistivity, thermal conductivity.

Annex H. Data Summary and Calculations

Table A1 contains the 24 experimental data points plotted in Figure 3. Table A2 is a summary of data fits by a least-squares method of these data. Set 1 (all data) yielded fits with average percent deviations of 0.65% and intercept (k_o) values for region 1 and 2. Set 1 provides a basis for the following comparisons.

Sets 2 and 3 are subsets of Set 1. Sets 2 and 3 show data for the PIMA Standard would be within 25 of the expectations for all data values for k_o. Table A2 show the predicted thermal performance (k and 1/k) values for Sets 2 and 3 are within 2% of the values for Set 1. Table A3 shows the predicted time-averaged thermal resistivity values for Set 1 for times of 1, 5 and 10 years. Data sets 2 and 3 agree to 1% with these values.

Example: PIMA Document
Saved as PIMADRF2

Date	Test No.	Specimen Age (days)	h (in)	k (Btu.in/h.ft ^ 2.F)	ln 100*k	t/h ^ 2 (d/in ^ 2)	Sqrt t/h ^ 2
	1	3	1.299	0.1268	2.5400	1.777	1.3332
	2	17	1.299	0.1239	2.5169	10.072	3.1736
	3	51.5	1.299	0.1315	2.5764	30.511	5.5237
	4	106.5	1.299	0.1322	2.5817	63.095	7.9433
	5	190	1.299	0.1418	2.6518	112.565	10.6096
	6	290	1.299	0.144	2.6672	171.809	13.1076
	7	463.5	1.299	0.1488	2.7000	274.598	16.5710
	8**	873.5	1.299	0.1577	2.7581	517.501	22.7486
	1	3	0.756	0.1206	2.4899	5.250	2.2914
	2	17	0.756	0.1286	2.5541	29.752	5.4546
	3	51.5	0.756	0.1396	2.6362	90.132	9.4938
	4	106.5	0.756	0.1476	2.6919	186.389	13.6524
	5**	190	0.756	0.154	2.7344	332.525	18.2353
	6**	290	0.756	0.1602	2.7738	507.539	22.5286
	7**	463.5	0.756	0.1634	2.7936	811.187	28.4813
	8**	873.5	0.756	0.1691	2.8279	1528.742	39.0991
	1	3	0.398	0.1322	2.5817	18.977	4.3563
	2	17	0.398	0.139	2.6319	107.537	10.3700
	3**	51.5	0.398	0.1502	2.7094	325.773	18.0492
	4**	106.5	0.398	0.156	2.7473	673.685	25.9554
	5**	190	0.398	0.1592	2.7676	1201.879	34.6681
	6**	290	0.398	0.1628	2.7899	1834.447	42.8304
	7**	463.5	0.398	0.1652	2.8046	2931.953	54.1475
	8**	873.5	0.398	0.1728	2.8495	5525.482	74.3336

TABLE A1. SUMMARY OF TEST DATA FOR CFC-11 BOARD. Apparent thermal conductivity (k) results are converted to the natural logarithm of 100*k. Specimen thickness (h) and age at time (t) of test are converted to values of (time) $^{1/2}$ divided by thickness. **: Data Point is in Region 2.

TABLE A2. SUMMARY OF DATA FITS BY A LEAST-SQUARES METHOD FOR SPECIMENS BLOWN WITH CFC-11 AND AGING AT 75°F.

1. All Data

Region	A, Btu*in/(h*ft²*°F)	B * 10^{-3}, $(in^2/d)^{1/2}$	Number of Data Points	Average Percent Deviation, (%)	Intercept, k_o Btu*in/(h*ft²*°F)	Percent Difference, %
1.	2.4942	13.427	13	0.65	0.1211	
2.	2.7084	2.003	11	0.65	0.1500	

2. 1.3 inch and 0.4 inch: All Data.

Region	A, Btu*in/(h*ft²*°F)	B * 10^{-3}, $(in^2/d)^{1/2}$	Number of Data Points	Average Percent Deviation, (%)	Intercept, k° Btu*in/(h*ft²*°F)	Percent Difference, %
1.	2.5102	11.723	9	0.49	0.1231	1.6
2.	2.6905	2.172	7	0.27	0.1474	-1.7

3. 1.3 inch and 0.4 inch: Data to 190 days.

Region	A, Btu*in/(h*ft²*°F)	B * 10^{-3}, $(in^2/d)^{1/2}$	Number of Data Points	Average Percent Deviation, (%)	Intercept, k° Btu*in/(h*ft²*°F)	Percent Difference, %
1.	2.5085	12.030	7	0.59	0.1299	1.5
2.	2.6501	3.482	3	0.16	0.1416	-5.6

TABLE A3. PREDICTED THERMAL PERFORMANCE FOR 1.5 INCH THICK FOAM BOARD BLOWN WITH CFC-11.

1. All Data

Property	Initial Value	One Year	Five Years	10 Years
k	0.1211	0.1437	0.1589	0.1627
1/k	8.25	6.958	6.295	6.146

2. 1.3 inch and 0.4 inch data.

Property	Initial Value	One Year	Five Years	10 Years
k	0.1231	0.1429, -0.6%	0.1568, -1.3%	0.1609, -1.1%
1/k	8.126	6.998	6.378	6.215

3. 1.3 inch and 0.4 inch data to 190 days.

Property	Initial Value	One Year	Five Years	10 Years
k	0.1275	0.1432, -0.4%	0.1560, -1.8%	0.1604, -1.4%
1/k	7.845	6.982	6.411	6.234

TABLE A4. PREDICTED TIME-AVERAGED THERMAL RESISTIVITY FOR 1.5 INCH THICK FOAM BLOWN WITH CFC-11.

1. All Data.

Time-Averaged Property	One Year	Five Years	Ten Years
k	0.1319	0.1438	0.1486
1/k	7.579	6.950	6.728

2. 1.3 inch and 0.4 inch: all data.

Time-Averaged Property	One Year	Five Years	Ten Years
k	0.1326	0.1432	0.1476
1/k	7.542, -0.49%	6.983, 0.47%	6.775, 0.70%

3. 1.3 inch and 0.4 inch: 190 day data.

Time-Averaged Property	One Year	Five Years	Ten Years
k	0.1326	0.1432	0.1476
1/k	7.541, -0.5%	7.003, 0.76%	6.78, 0.77%

TABLE A5. COMPARISON OF THICKNESS IN INCHES OF INDIVIDUAL AND STACKED BOARDS DETERMINED AT ORNL USING:

(A) COORDINATE MEASURING MACHINE, AND
(B) CALIBRATED HEAT FLOW METER APPARATUS (HFMA).

A. THICKNESS BY CALIBRATED MEASURING MACHINE.

BOARD 1	BOARD 2	BOARD 3	BOARD 4	STACKED BOARDS 1-2-3-4
0.347	0.334	0.334	0.323	1.339
0.329	0.341	0.321	0.341	1.323
0.329	0.325	0.313	0.343	1.303
0.344	0.331	0.344	0.315	1.330
0.341	0.340	0.340	0.340	1.356
0.338	0.320	0.337	0.331	1.323
0.344	0.331	0.343	0.315	1.329
0.341	0.340	0.339	0.339	1.353
0.340	0.324	0.337	0.330	1.325
0.334	0.343	0.333	0.332	1.334
0.324	0.345	0.313	0.343	1.318
0.327	0.335	0.317	0.348	1.319
0.3365-AVG	0.3341-AVG	0.33175-AVG	0.3333-AVG	1.3293-AVG
			1.3356-SUM 1-2-3-4	

B. CALIBRATED HFMA ON STACKED BOARDS 1-2-3-4: 1.3150.

C. RATIO OF MEASURED THICKNESS TO HFMA THICKNESS:
  A. AVERAGE BOARDS 1-2-3-4 / HFMA: 1.0157
  B. SUM 1-2-3-4 / HFMA: 1.0157

APPENDIX C

PROCEDURE FOR INTERLABORATORY COMPARISON

INTERLABORATORY COMPARISON

ON

PIMA STANDARD FOR ESTIMATING THE LONG-TERM
THERMAL PERFORMANCE OF
UNFACED, RIGID, CLOSED-CELL POLYISOCYANURATE FOAM INSULATION

PROCEDURE

1) ORNL will provide each participant five pieces of sliced polyisocyanurate foam. Specimen #1 will be approximately 1.35 in. thick. Specimen #2 will be comprised of four thin slices (0.35 in.) that will be stacked for test purposes. Each piece of foam is marked with a number in the corner of one major surface for identification and orientation purposes.

2) ORNL will provide the following:

   a) Date of foam manufacture
   b) Date of slicing (t=0)
   c) Initial thickness of both test specimens.

3) Participant shall ensure that their heat flow meter apparatus is calibrated and that it is available for the required tests over the 180 day time period.

4) Determine the thickness, area and weight of each slice of foam. Calculate the volume and density.

5) Specimen #1 is the 1.35 in. thick foam. Load the specimen so that the mark is facing up and is positioned at the left-back corner of the apparatus.

6) Specimen #2 is comprised of the four thin slices stacked so that the marked corners are facing up and toward the left and back as you face the apparatus. Stack the foam pieces so that the smallest number is at the top and the largest number is on the bottom of the stack. This same orientation is required for all tests.

7) Test specimens #1 and 2 according to the following schedule.

Test 1	01-07-94 & 01-10-94	(Immediately after receiving)
Test 2	01-12-94	
Test 3	01-19-94	
Test 4	01-26-94	
Test 5	02-02-94	
Test 6	03-16-94	
Test 7	04-13-94	

Test 5	02-02-94
Test 6	03-16-94
Test 7	04-13-94
Test 8	05-11-94
Test 9	06-08-94
Test 10	07-06-94

It is important that you test both specimens on the same day of the week and follow the schedule shown above.

8) Perform all tests at a mean temperature of 75$\pm$4°F with a temperature difference of 40 or 50°F.

9) Record the tests results in Tables 1 and 2. Supply the information requested on the Laboratory Report Form.

10) Continue the test sequence until ten tests are completed on both specimens.

11) Analysis of the Data: Fit the data following the example in ANNEX B. Region 1 should consist of data from tests 1-5 for the stacked thin sliced specimen and from all ten tests for the thick specimen. Region 2 should contain only data from tests 6-10 for the stacked thin slices. Perform a least squares method fit on these data sets. Use this analysis to complete Table 3, Table 4 and Section 7.9. Additional information will be provided in the near future.

12) Between tests all slices shall be stored at 75$\pm$4°F and 50$\pm$5% RH such that the major surfaces are exposed to the air. It is suggested that you allow each slice to rest vertically on one edge. Avoid face-to-face contact with adjacent slices. At ORNL, I rest slices loosely on their edges with cardboard spacers between slices (like a row of books).

13) Handle the slices with reasonable care to avoid tears and breaks.

14) Data may be reported in English or SI units but the analyses will be based on English units for the present.

15) If there are any questions call R. S. Graves at (615) 574-5978.

APPENDIX D

ANALYSIS BY ORNL OF DATA OBTAINED FROM PARTICIPANTS

Table D-1

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant A: Specimens # 3/18/19/20/21

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
12/09/93	01-0001		70.00	1	10		1.3200	1.3200		0.1360	2.3957	2.6101
12/15/93	01-0002		70.00	1	16		1.3100	1.3100		0.1350	3.0534	2.6027
12/22/93	01-0003		70.00	1	23		1.3100	1.3100		0.1390	3.6609	2.6319
12/30/93	01-0004		70.00	1	31		1.3200	1.3200		0.1420	4.2180	2.6532
01/05/94	01-0005		70.00	1	37		1.3200	1.3200		0.1450	4.6082	2.6741
02/16/94	01-0006		70.00	1	79		1.3100	1.3100		0.1570	6.7849	2.7537
03/16/94	01-0007		70.00	1	107		1.3200	1.3200		0.1610	7.8364	2.7788
04/13/94	01-0008		70.00	1	135		1.3100	1.3100		0.1660	8.8694	2.8094
05/11/94	01-0009		70.00	1	163		1.3200	1.3200		0.1650	9.6721	2.8034
06/08/94	01-0010		70.00	1	191		1.3200	1.3200		0.1720	10.4699	2.8449
12/09/93	02-0001		70.00	4	10		1.2600	0.3150		0.1730	10.0390	2.8507
12/15/93	02-0002		70.00	4	16		1.2500	0.3125		0.1750	12.8000	2.8622
12/22/93	02-0003		70.00	4	23		1.2600	0.3150		0.1810	15.2249	2.8959
12/30/93	02-0004		70.00	4	31		1.2600	0.3150		0.1860	17.6754	2.9232
01/05/94	02-0005		70.00	4	37		1.2600	0.3150		0.1860	19.3104	2.9232
02/16/94	02-0006		70.00	4	79		1.2700	0.3175		0.1900	27.9943	2.9444
03/16/94	02-0007		70.00	4	107		1.2700	0.3175		0.1910	32.5798	2.9497
04/13/94	02-0008		70.00	4	135		1.2700	0.3175		0.1940	36.5951	2.9653
05/11/94	02-0009		70.00	4	163		1.2700	0.3175		0.1910	40.2115	2.9497
06/08/94	02-0010		70.00	4	191		1.2700	0.3175		0.1960	43.5284	2.9755

Figure D-4

PIMA RR (HCFC-141b)
BOARDS # 3 / 18, 19, 20, 21.

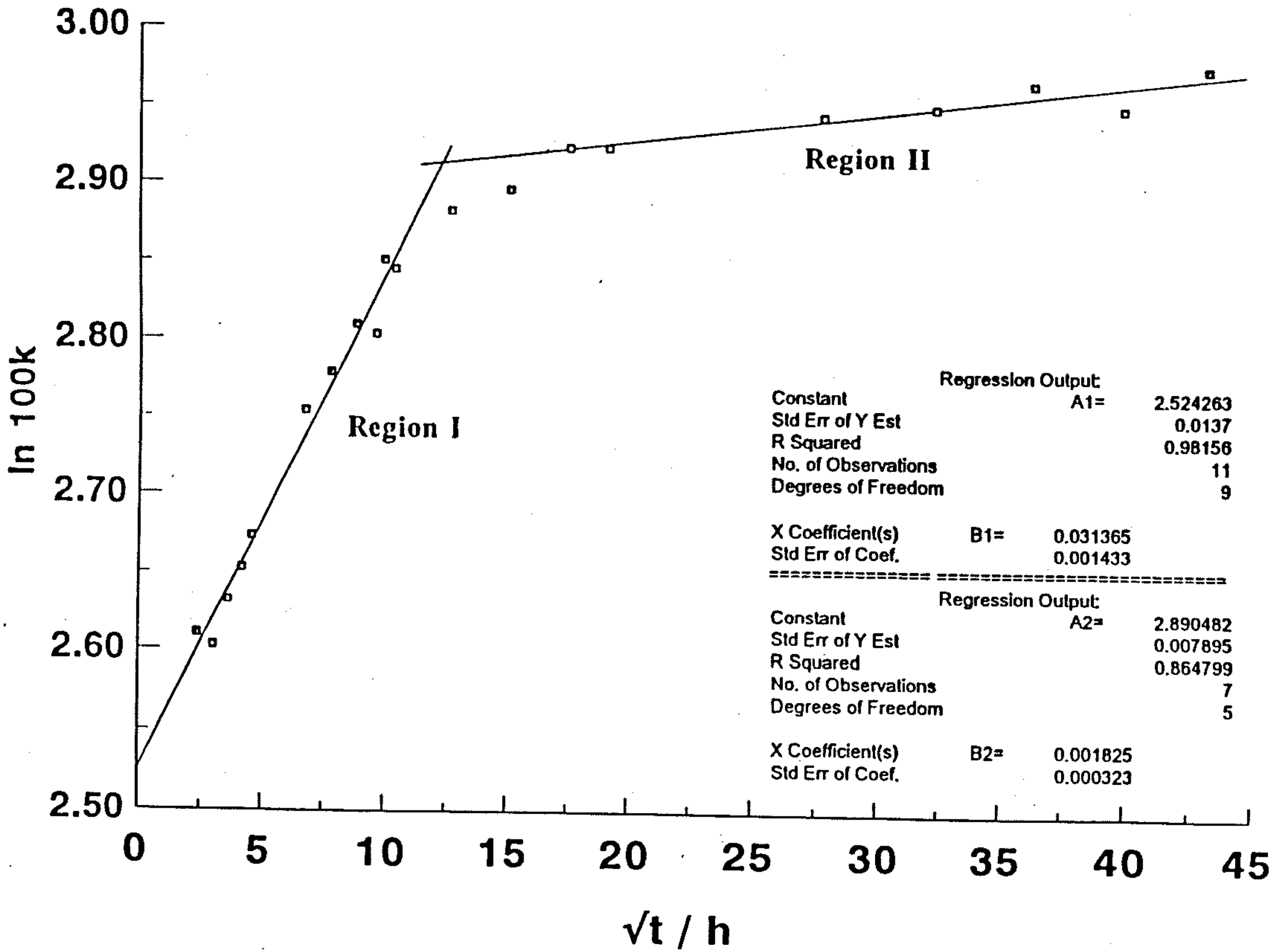


Table D-2

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participate B: Specimens #1/10/11/12/13

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (In)	Avg. Aging Thickness (In)	k (w/m.K)	k (Btu.In/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/In	Y In 100k (Btu.In/h.ft^2.F)
12/13/93	00-0001		75.00	1	14		1.2840	1.2840		0.1382	2.9141	2.6261
12/15/93	00-0002		75.00	1	16		1.2840	1.2840		0.1382	3.1153	2.6261
12/22/93	00-0003		75.00	1	23		1.2840	1.2840		0.1417	3.7351	2.6511
12/29/93	00-0004		75.00	1	30		1.2840	1.2840		0.1444	4.2658	2.6700
01/05/94	00-0005		75.00	1	37		1.2830	1.2830		0.1466	4.7410	2.6851
02/16/94	00-0006		75.00	1	79		1.2830	1.2830		0.1556	6.9277	2.7447
03/16/94	00-0007		75.00	1	107		1.2840	1.2840		0.1633	8.0561	2.7930
04/13/94	00-0008		75.00	1	135		1.2880	1.2880		0.1677	9.0209	2.8196
05/11/94	00-0009		75.00	1	163		1.2900	1.2900		0.1705	9.8970	2.8362
06/13/94	00-0010		75.00	1	196		1.2900	1.2900		0.1742	10.8527	2.8576
12/13/93	00-0001		75.00	4	14		1.2440	0.3110		0.1805	12.0311	2.8931
12/15/93	00-0002		75.00	4	16		1.2440	0.3110		0.1816	12.8617	2.8992
12/22/93	00-0003		75.00	4	23		1.2440	0.3110		0.1835	15.4207	2.9096
12/29/93	00-0004		75.00	4	30		1.2430	0.3108		0.1867	17.6258	2.9269
01/05/94	00-0005		75.00	4	37		1.2390	0.3098		0.1894	19.6377	2.9413
02/16/94	00-0006		75.00	4	79		1.2420	0.3105		0.1945	28.6254	2.9678
03/16/94	00-0007		75.00	4	107		1.2400	0.3100		0.1960	33.3680	2.9755
04/13/94	00-0008		75.00	4	135		1.2460	0.3115		0.1985	37.3000	2.9882
05/11/94	00-0009		75.00	4	163		1.2470	0.3118		0.1990	40.9532	2.9907
06/13/94	00-0010		75.00	4	196		1.2450	0.3113		0.2001	44.9799	2.9962

Figure D-2

PIMA RR (HCFC-141b)

Figure D-2

PIMA RR (HCFC-141b)
BOARDS # 1 / 10, 11, 12, 13.

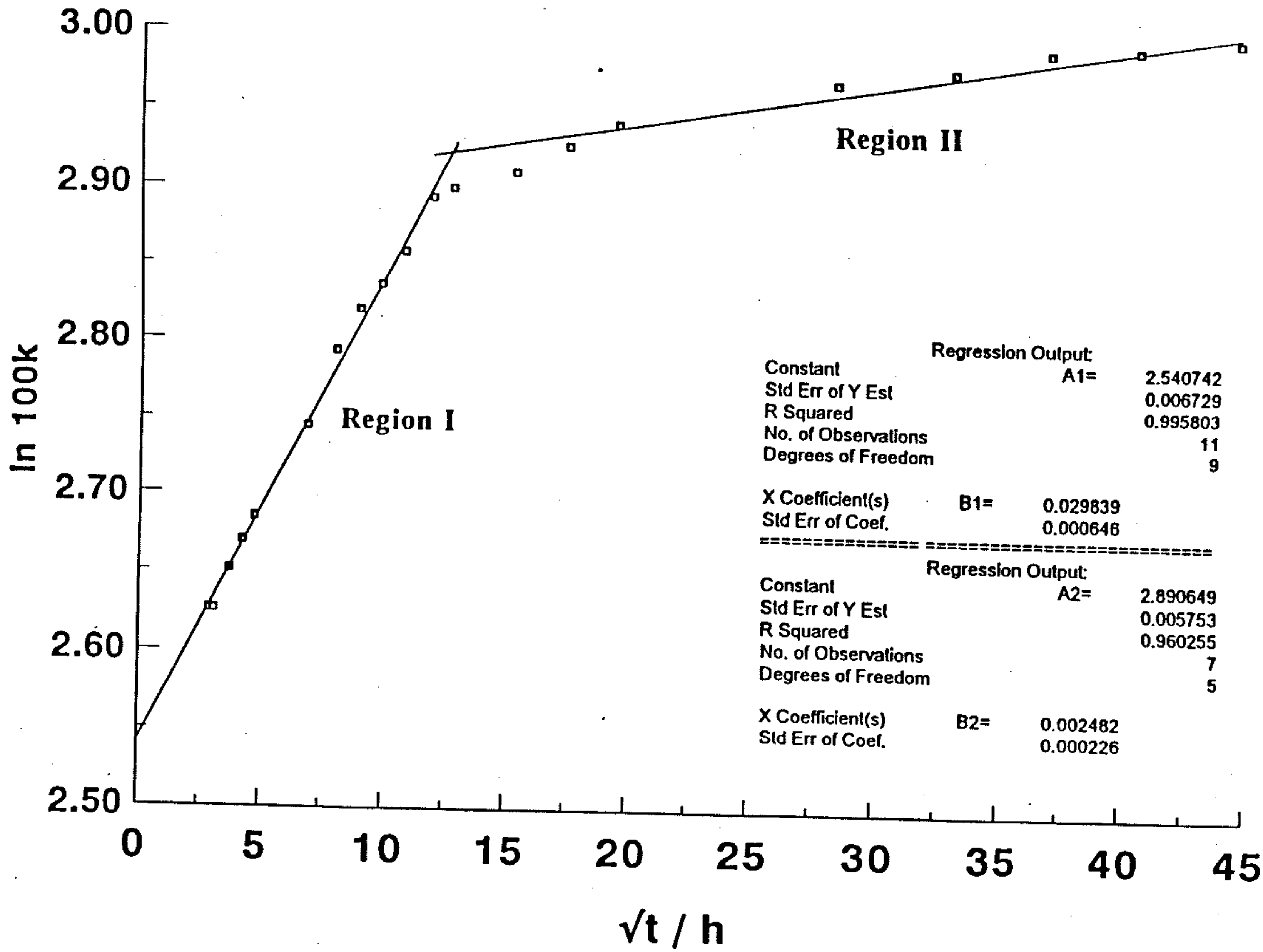


Table D-3

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant C: Specimens # 49/75/76/77/78

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
01/10/94	00-0000		75.00	1	7		1.3500	1.3500		0.1370	1.9598	2.6174
01/12/94	00-0000		75.00	1	9		1.3500	1.3500		0.1390	2.2222	2.6319
01/19/94	00-0000		75.00	1	16		1.3500	1.3500		0.1400	2.9630	2.6391
01/26/94	00-0000		75.00	1	23		1.3500	1.3500		0.1420	3.5525	2.6532
02/02/94	00-0000		75.00	1	30		1.3500	1.3500		0.1440	4.0572	2.6672
03/16/94	00-0000		75.00	1	72		1.3600	1.3600		0.1480	6.2392	2.6946
04/13/94	00-0000		75.00	1	100		1.3600	1.3600		0.1580	7.3529	2.7600
05/11/94	00-0000		75.00	1	128		1.3500	1.3500		0.1600	8.3805	2.7726
06/08/94	00-0000		75.00	1	156		1.3600	1.3600		0.1630	9.1838	2.7912
07/06/94	00-0000		75.00	1	184		1.3600	1.3600		0.1680	9.9740	2.8214
01/10/94	00-0000		75.00	4	7		1.5000	0.3750		0.1670	7.0553	2.8154
01/12/94	00-0000		75.00	4	9		1.5000	0.3750		0.1700	8.0000	2.8332
01/19/94	00-0000		75.00	4	16		1.5000	0.3750		0.1750	10.6667	2.8622
01/26/94	00-0000		75.00	4	23		1.5100	0.3775		0.1800	12.7042	2.8904
02/02/94	00-0000		75.00	4	30		1.5100	0.3775		0.1830	14.5092	2.9069
03/16/94	00-0000		75.00	4	72		1.5200	0.3800		0.1830	22.3297	2.9069
04/13/94	00-0000		75.00	4	100		1.5100	0.3775		0.1930	26.4901	2.9601
05/11/94	00-0000		75.00	4	128		1.5100	0.3775		0.1950	29.9701	2.9704
06/08/94	00-0000		75.00	4	156		1.5200	0.3800		0.1980	32.8684	2.9857
07/06/94	00-0000		75.00	4	184		1.5200	0.3800		0.1990	35.6965	2.9907

Figure D-3

PIMA RR (HCFC-141b)
BOARDS # 49 / 75, 76, 77, 78.

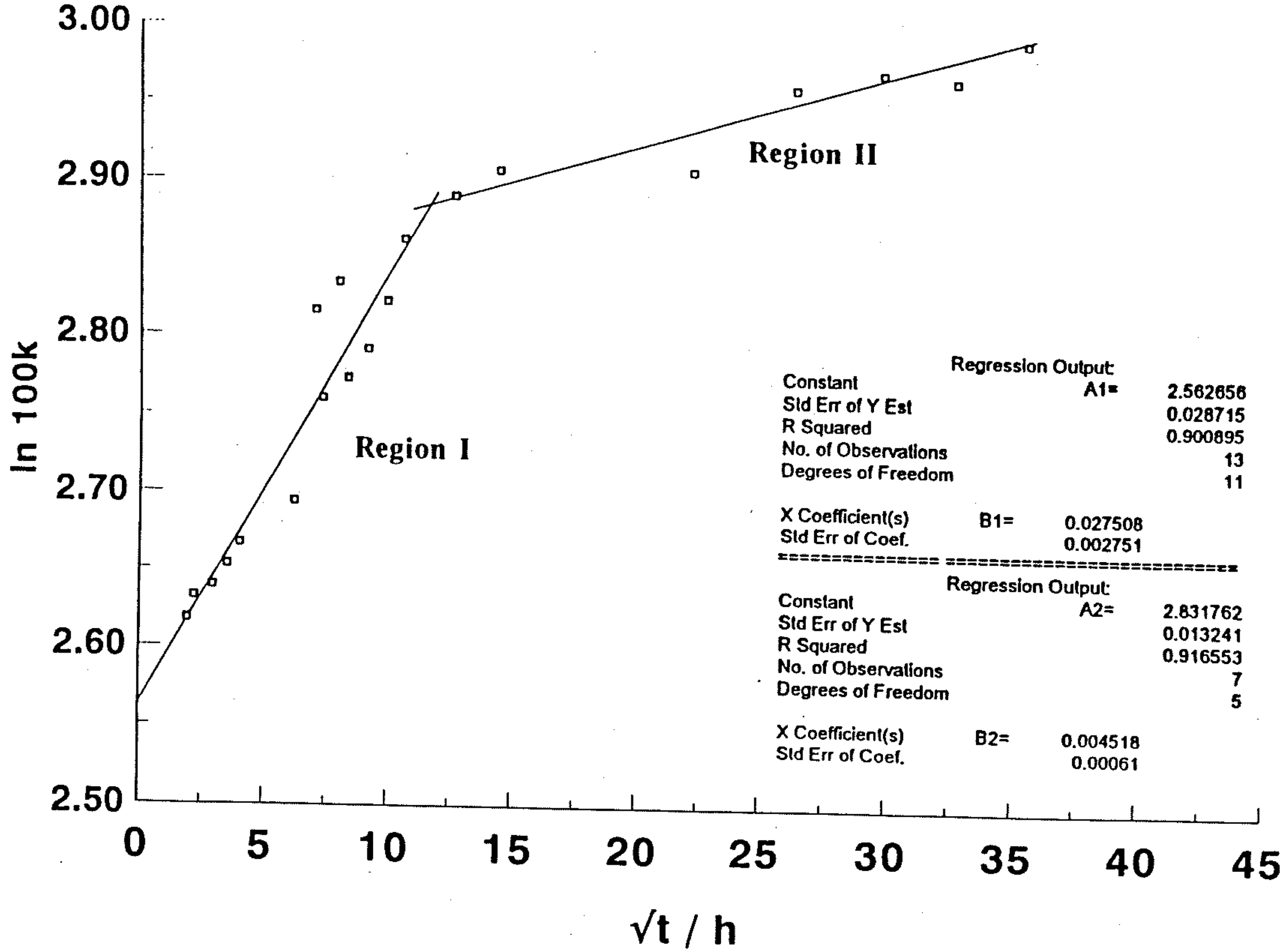


Table D-4

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant D: Specimens # 47/67/68/69/70

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
01/10/94	942T-3A		74.70	4	7		1.5040	0.3760		0.1620	7.0366	2.7850
01/12/94	942T-3A		74.90	4	9		1.5030	0.3758		0.1660	7.9840	2.8094
01/19/94	942T-3A		75.00	4	16		1.5030	0.3758		0.1740	10.6454	2.8565
01/26/94	942T-3A		74.80	4	23		1.5010	0.3753		0.1760	12.7804	2.8679
02/02/94	942T-3A		75.30	4	30		1.5040	0.3760		0.1800	14.5671	2.8904
03/16/94	942T-3A		74.70	4	72		1.5060	0.3765		0.1880	22.5373	2.9339
04/13/94	942T-3A		75.00	4	100		1.5060	0.3765		0.1880	26.5604	2.9339
05/11/94	942T-3A		74.70	4	128		1.5060	0.3765		0.1890	30.0497	2.9392
06/08/94	942T-3A		74.80	4	156		1.5070	0.3768		0.1890	33.1519	2.9392
07/12/94	942T-3A		74.90	4	190		1.5070	0.3768		0.1920	36.5867	2.9549
01/10/94	t94t-23		75.10	1	7		1.3350	1.3350		0.1470	1.9818	2.6878
01/12/94	t94t-23		74.90	1	9		1.3400	1.3400		0.1400	2.2388	2.6391
01/19/94	t94t-23		75.00	1	16		1.3370	1.3370		0.1520	2.9918	2.7213
01/26/94	t94t-23		74.80	1	23		1.3370	1.3370		0.1500	3.5870	2.7081
02/02/94	t94t-23		74.90	1	30		1.3380	1.3380		0.1480	4.0936	2.6946
03/16/94	t94t-23		74.80	1	72		1.3420	1.3420		0.1590	6.3229	2.7663
04/13/94	t94t-23		74.80	1	100		1.3420	1.3420		0.1670	7.4516	2.8154
05/11/94	t94t-23		74.90	1	128		1.3400	1.3400		0.1730	8.4431	2.8507
06/08/94	t94t-23		74.80	1	156		1.3380	1.3380		0.1730	9.3348	2.8507
07/12/94	t94t-23		75.10	1	190		1.3380	1.3380		0.1710	10.3020	2.8391

Figure D-4

PIMA RR (HCFC-141b)
BOARDS # 47 / 67, 68, 69, 70.

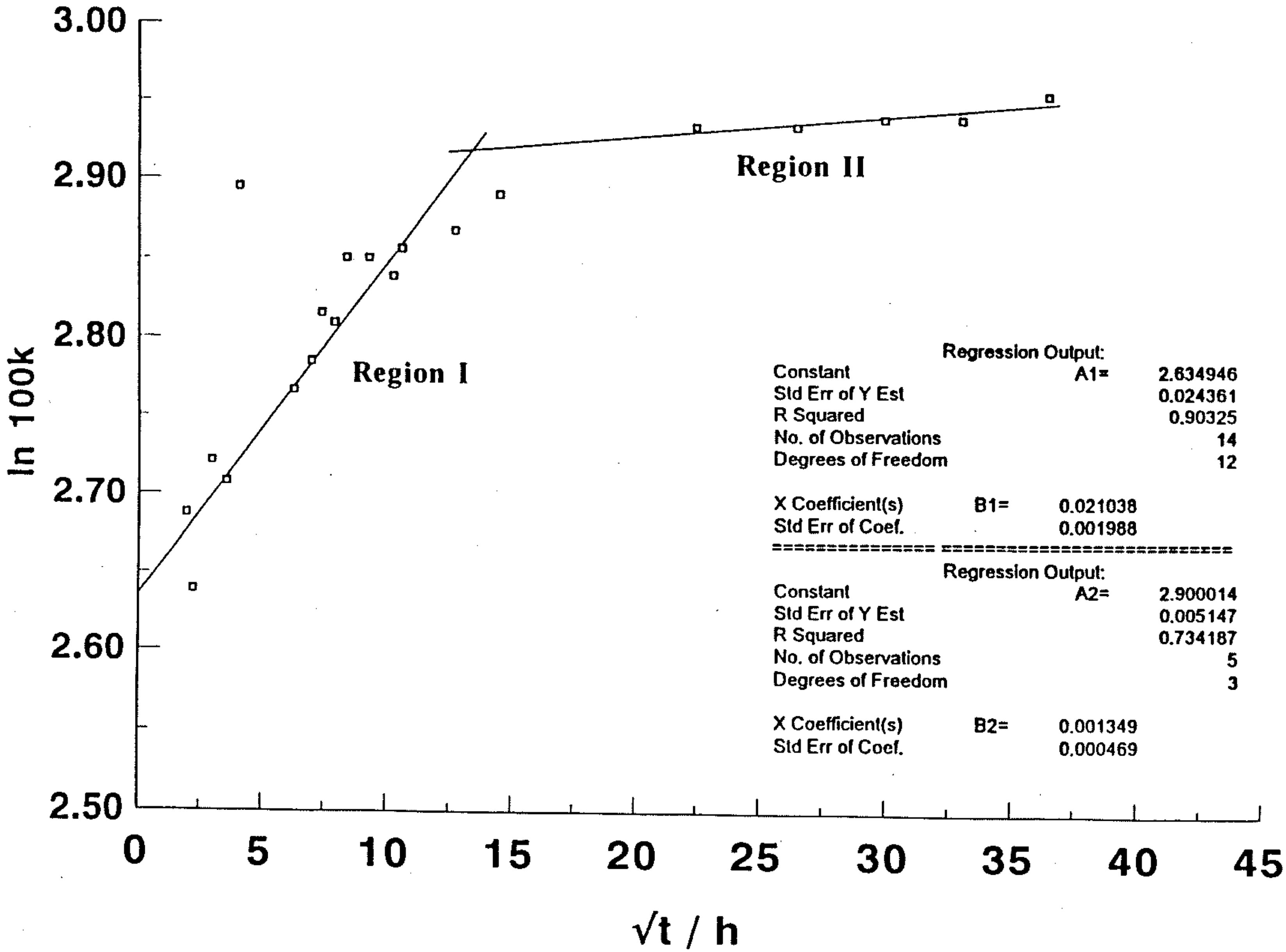


Table D-5

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant E: Specimens # 6/30/31/32/33

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
12/08/93	00-0000		75.00	1	9	3.230	1.2717	1.2717		0.1370	2.3591	2.6174
12/15/93	00-0000		75.00	1	16	3.230	1.2717	1.2717		0.1380	3.1455	2.6247
12/22/93	00-0000		75.00	1	23	3.230	1.2717	1.2717		0.1390	3.7713	2.6319
12/29/93	00-0000		75.00	1	30	3.230	1.2717	1.2717		0.1420	4.3072	2.6532
01/05/94	00-0000		75.00	1	37	3.230	1.2717	1.2717		0.1430	4.7833	2.6603
02/16/94	00-0000		75.00	1	79	3.230	1.2717	1.2717		0.1560	6.9895	2.7473
03/16/94	00-0000		75.00	1	107	3.230	1.2717	1.2717		0.1600	8.1344	2.7726
04/13/94	00-0000		75.00	1	135	3.230	1.2717	1.2717		0.1630	9.1369	2.7912
05/11/94	00-0000		75.00	1	162	3.230	1.2717	1.2717		0.1680	10.0090	2.8214
06/08/94	00-0000		75.00	1	190	3.230	1.2717	1.2717		0.1710	10.8395	2.8391
12/08/93	00-0000		75.00	4	9	3.170	1.2480	0.3120		0.1700	9.6151	2.8332
12/15/93	00-0000		75.00	4	16	3.150	1.2402	0.3100		0.1740	12.9016	2.8565
12/22/93	00-0000		75.00	4	23	3.150	1.2402	0.3100		0.1790	15.4685	2.8848
12/29/93	00-0000		75.00	4	30	3.150	1.2402	0.3100		0.1800	17.6662	2.8904
01/05/94	00-0000		75.00	4	37	3.180	1.2520	0.3130		0.1830	19.4342	2.9069
02/16/94	00-0000		75.00	4	79	3.180	1.2520	0.3130		0.1870	28.3975	2.9285
03/16/94	00-0000		75.00	4	107	3.180	1.2520	0.3130		0.1890	33.0490	2.9392
04/13/94	00-0000		75.00	4	135	3.180	1.2520	0.3130		0.1890	37.1222	2.9392
05/11/94	00-0000		75.00	4	162	3.180	1.2520	0.3130		0.1920	40.6653	2.9549
06/08/94	00-0000		75.00	4	190	3.150	1.2402	0.3100		0.1930	44.4590	2.9601

Figure D-5

PIMA RR (HCFC-141b)
BOARDS # 6 / 30, 31, 32, 33.

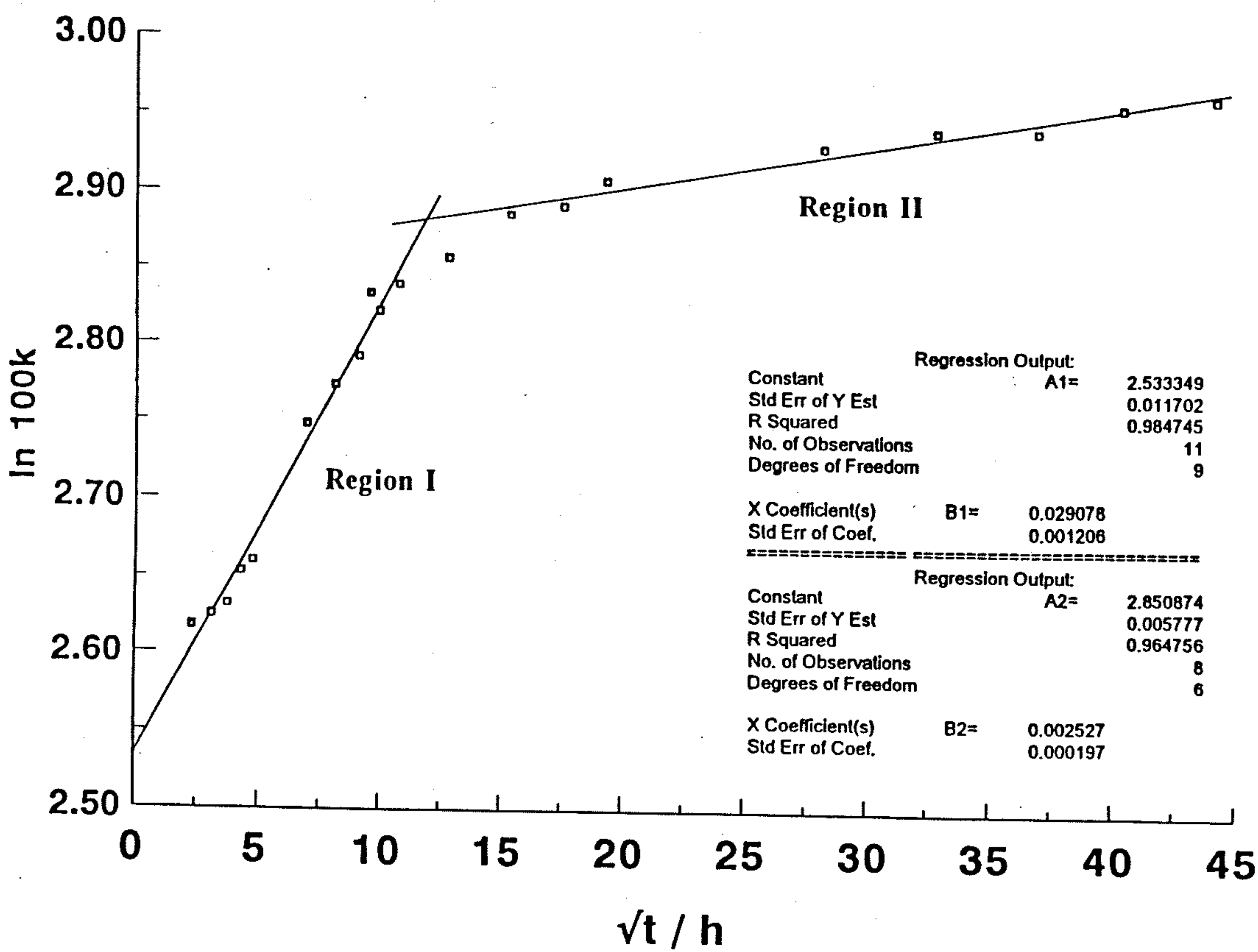


Table D-6

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant F: Specimens # 5/26/27/28/29

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (In)	Avg. Aging Thickness (In)	k (w/m.K)	k (Btu.In/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/In	Y ln 100k (Btu.In/h.ft^2.F)
12/10/93	00-0001		75.10	1	11		1.3250	1.3250		0.1336	2.5031	2.5923
12/15/93	00-0002		75.13	1	16		1.3240	1.3240		0.1364	3.0211	2.6130
12/22/93	00-0003		75.07	1	23		1.3230	1.3230		0.1398	3.6250	2.6376
12/29/93	00-0004		75.04	1	30		1.3210	1.3210		0.1431	4.1463	2.6610
01/05/94	00-0005		75.04	1	37		1.3220	1.3220		0.1469	4.6012	2.6872
02/16/94	00-0006		75.19	1	79		1.3220	1.3220		0.1563	6.7233	2.7492
03/16/94	00-0007		74.80	1	107		1.3220	1.3220		0.1625	7.8246	2.7881
04/13/94	00-0008		75.58	1	135		1.3270	1.3270		0.1672	8.7558	2.8166
05/11/94	00-0009		76.23	1	163		1.3250	1.3250		0.1708	9.6356	2.8379
06/08/94	00-0010		75.80	1	191		1.3300	1.3300		0.1715	10.3912	2.8420
12/10/93	02-0001		74.74	4	11		1.2630	0.3158		0.1671	10.5040	2.8160
12/15/93	02-0002		75.52	4	16		1.2630	0.3158		0.1731	12.6683	2.8513
12/27/93	02-0003		74.98	4	23		1.2610	0.3153		0.1775	15.2128	2.8764
12/29/93	02-0004		75.01	4	30		1.2600	0.3150		0.1807	17.3880	2.8943
01/05/94	02-0005		74.87	4	37		1.2610	0.3153		0.1839	19.2950	2.9118
02/16/94	02-0006		74.95	4	79		1.2610	0.3153		0.1886	28.1941	2.9370
03/16/94	02-0007		75.13	4	107		1.2610	0.3153		0.1894	32.8123	2.9413
04/13/94	02-0008		75.45	4	135		1.2650	0.3163		0.1915	36.7398	2.9523
05/11/94	02-0009		75.52	4	163		1.2630	0.3158		0.1920	40.4343	2.9549
06/08/94	02-0010		75.51	4	191		1.2700	0.3175		0.1921	43.5284	2.9554

Figure D-6

Figure D-6

PIMA RR (HCFC-141b)
BOARDS # 5 / 26, 27, 28, 29.

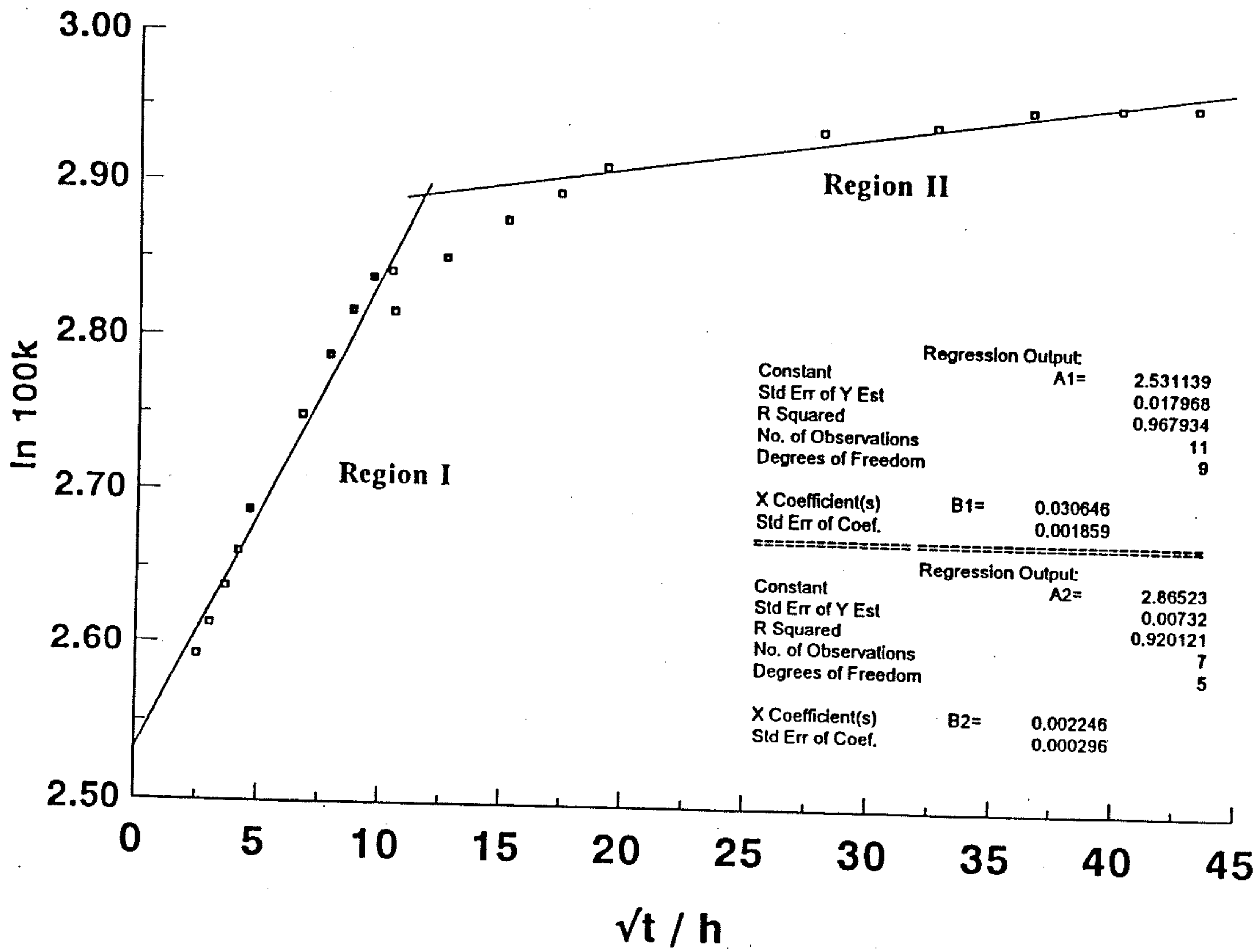


Table D-7

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant G: Specimens # 2/14/15/16/17

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
12/08/93	1	23.00	73.40	4	9	3.210	1.2638	0.3159	0.02480	0.1719	9.4953	2.8446
12/15/93	2	23.00	73.40	4	16	3.210	1.2638	0.3159	0.02620	0.1816	12.6604	2.8995
12/22/93	3	24.00	75.20	4	23	3.210	1.2638	0.3159	0.02660	0.1844	15.1793	2.9146
12/29/93	4	24.00	75.20	4	30	3.210	1.2638	0.3159	0.02710	0.1879	17.3360	2.9332
01/05/94	5	24.00	75.20	4	37	3.210	1.2638	0.3159	0.02720	0.1886	19.2526	2.9369
02/18/94	6	25.00	77.00	4	81	3.210	1.2638	0.3159	0.02800	0.1941	28.4860	2.9659
03/16/94	7	24.00	75.20	4	107	3.210	1.2638	0.3159	0.02800	0.1941	32.7401	2.9659
04/13/94	8	24.00	75.20	4	145	3.210	1.2638	0.3159	0.02810	0.1948	38.1130	2.9695
05/11/94	9	24.00	75.20	4	173	3.210	1.2638	0.3159	0.02760	0.1914	41.6305	2.9515
06/08/94	10	24.00	75.20	4	201	3.210	1.2638	0.3159	0.02780	0.1927	44.8732	2.9587
12/08/93	1	23.00	73.40	1	9	3.300	1.2992	1.2992	0.01930	0.1338	2.3091	2.5938
12/15/93	2	23.00	73.40	1	16	3.300	1.2992	1.2992	0.02000	0.1387	3.0788	2.6294
12/22/93	3	23.00	73.40	1	23	3.300	1.2992	1.2992	0.02060	0.1428	3.6913	2.6590
12/29/93	4	23.00	73.40	1	30	3.300	1.2992	1.2992	0.02070	0.1435	4.2158	2.6638
01/05/94	5	23.00	73.40	1	37	3.300	1.2992	1.2992	0.02120	0.1470	4.6819	2.6877
02/18/94	6	25.00	77.00	1	81	3.300	1.2992	1.2992	0.02280	0.1581	6.9273	2.7605
03/16/94	7	24.00	75.20	1	107	3.300	1.2992	1.2992	0.02320	0.1608	7.9618	2.7779
04/13/94	8	24.00	75.20	1	145	3.300	1.2992	1.2992	0.02410	0.1671	9.2684	2.8159
05/11/94	9	24.00	75.20	1	173	3.300	1.2992	1.2992	0.02400	0.1664	10.1238	2.8118
06/08/94	10	24.00	75.20	1	201	3.300	1.2992	1.2992	0.02440	0.1692	10.9123	2.8283

Figure D-7

PIMA RR (HCFC-141b)
BOARDS # 2 / 14, 15, 16, 17.

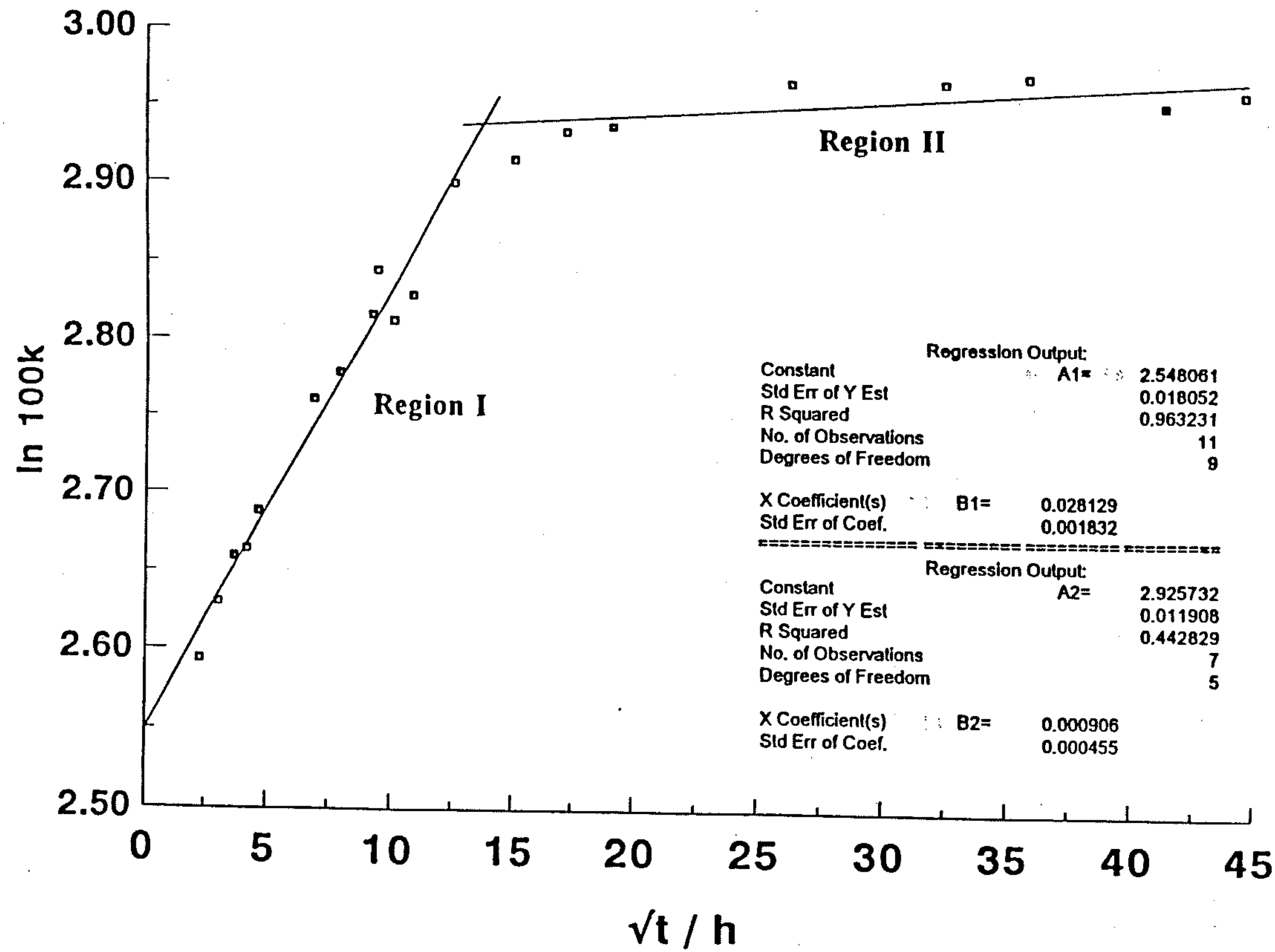


Table D-8

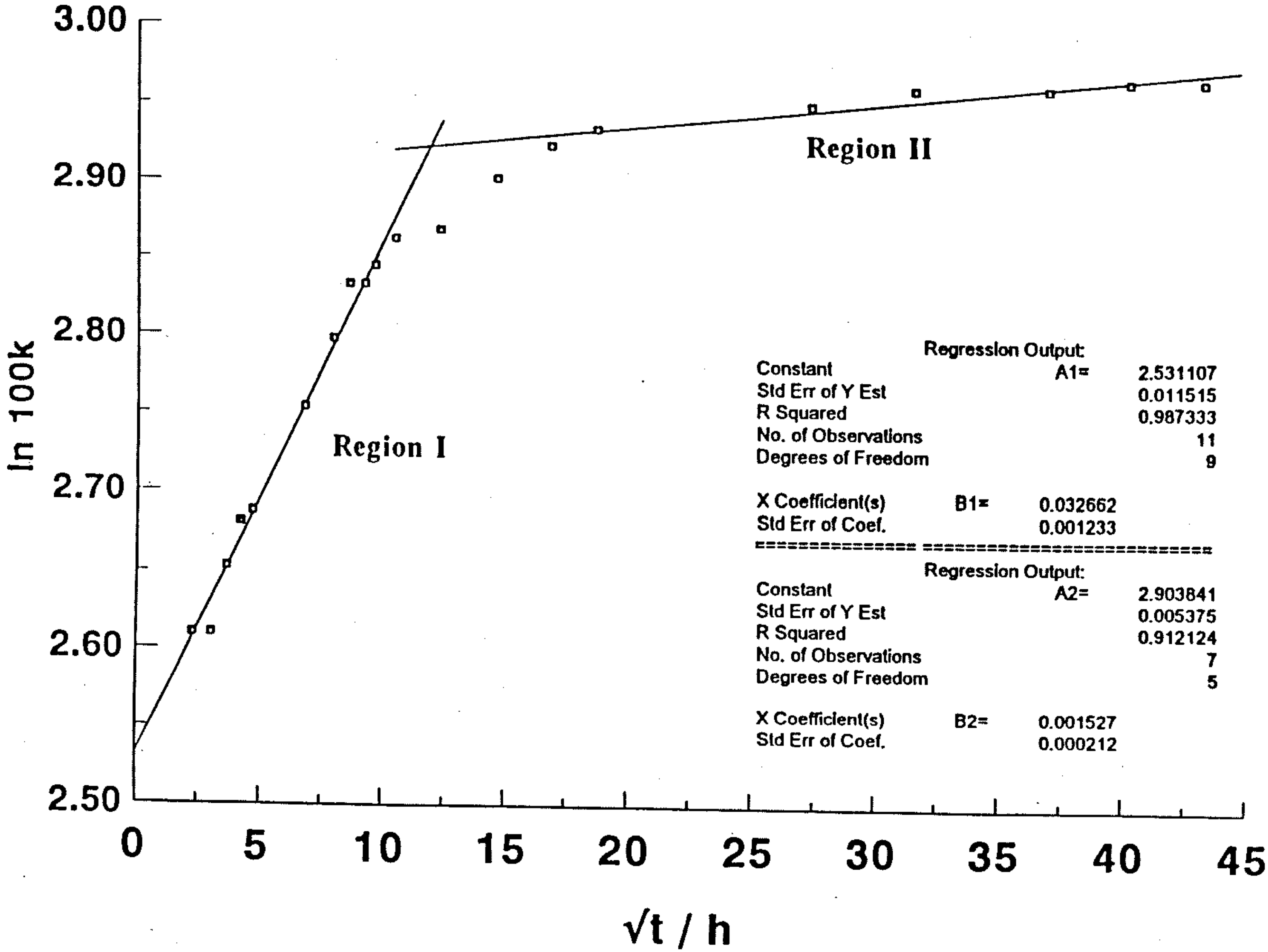
PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant H: Specimens # 4/22/23/24/25

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
12/08/93	00-0000		32.00	1	9		1.2900	1.2900		0.1360	2.3256	2.6101
12/15/93	00-0000		32.00	1	16		1.3000	1.3000		0.1360	3.0769	2.6101
12/22/93	00-0000		32.00	1	23		1.3000	1.3000		0.1420	3.6891	2.6532
12/29/93	00-0000		32.00	1	30		1.2900	1.2900		0.1460	4.2459	2.6810
01/05/94	00-0000		32.00	1	37		1.2900	1.2900		0.1470	4.7153	2.6878
02/16/94	00-0000		32.00	1	79		1.3000	1.3000		0.1570	6.8371	2.7537
03/16/94	00-0000		32.00	1	107		1.2900	1.2900		0.1640	8.0187	2.7973
04/13/94	00-0000		32.00	1	135		1.3400	1.3400		0.1700	8.6709	2.8332
05/11/94	00-0000		32.00	1	163		1.3120	1.3120		0.1720	9.7311	2.8449
06/08/94	00-0000		32.00	1	191		1.3090	1.3090		0.1750	10.5579	2.8622
12/08/93	00-0000		32.00	4	9		1.2900	0.3225		0.1700	9.3023	2.8332
12/15/93	00-0000		32.00	4	16		1.2900	0.3225		0.1760	12.4031	2.8679
12/22/93	00-0000		32.00	4	23		1.3000	0.3250		0.1820	14.7564	2.9014
12/29/93	00-0000		32.00	4	30		1.2900	0.3225		0.1860	16.9836	2.9232
01/05/94	00-0000		32.00	4	37		1.2900	0.3225		0.1880	18.8613	2.9339
02/16/94	00-0000		32.00	4	79		1.2900	0.3225		0.1910	27.5603	2.9497
03/16/94	00-0000		32.00	4	107		1.3000	0.3250		0.1930	31.8279	2.9601
04/13/94	00-0000		32.00	4	135		1.2500	0.3125		0.1930	37.1806	2.9601
05/11/94	00-0000		32.00	4	163		1.2600	0.3150		0.1940	40.5306	2.9653
06/08/94	00-0000		32.00	4	191		1.2690	0.3173		0.1940	43.5627	2.9653

Figure D-8

Figure D-8

PIMA RR (HCFC-141b)
BOARDS # 4 / 22, 23, 24, 25.



Regression Output:			
Constant		A1=	2.531107
Std Err of Y Est			0.011515
R Squared			0.987333
No. of Observations			11
Degrees of Freedom			9
X Coefficient(s)	B1=	0.032662	
Std Err of Coef.		0.001233	

Regression Output:			
Constant		A2=	2.903841
Std Err of Y Est			0.005375
R Squared			0.912124
No. of Observations			7
Degrees of Freedom			5
X Coefficient(s)	B2=	0.001527	
Std Err of Coef.		0.000212	

Table D-9

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant I: Specimens #45/56/58/59/61

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
01/12/94	1	24.00	75.20	4	9	3.649	1.4366	0.3592	0.02527	0.1752	8.3530	2.8633
01/19/94	2	24.00	75.20	4	16	3.653	1.4382	0.3595	0.02637	0.1828	11.1251	2.9059
01/26/94	3	24.00	75.20	4	23	3.651	1.4374	0.3594	0.02688	0.1864	13.3458	2.9251
02/02/94	4	24.00	75.20	4	30	3.648	1.4362	0.3591	0.02726	0.1890	15.2546	2.9391
02/09/94	5	24.00	75.20	4	37	3.654	1.4386	0.3596	0.02756	0.1911	16.9132	2.9501
02/16/94	6	24.00	75.20	4	44	3.653	1.4382	0.3595	0.02761	0.1914	18.4489	2.9519
02/23/94	7	24.00	75.20	4	51	3.653	1.4382	0.3595	0.02777	0.1925	19.8623	2.9577
03/02/94	8	24.00	75.20	4	58	3.650	1.4370	0.3593	0.02789	0.1934	21.1990	2.9620
03/09/94	9	24.00	75.20	4	65	3.653	1.4382	0.3595	0.02800	0.1941	22.4234	2.9659
04/20/94	10	24.00	75.20	4	107	3.651	1.4374	0.3594	0.02838	0.1968	28.7855	2.9794
05/20/94	11	24.00	75.20	4	137	3.658	1.4402	0.3600	0.02857	0.1981	32.5095	2.9861
06/08/94	12	24.00	75.20	4	156	3.658	1.4402	0.3600	0.02844	0.1972	34.6906	2.9815
07/13/94	13	24.00	75.20	4	191	3.657	1.4398	0.3599	0.02849	0.1975	38.3960	2.9833
01/12/94	1	24.00	75.20	1	9	3.388	1.3339	1.3339	0.02050	0.1421	2.2491	2.6541
01/19/94	2	24.00	75.20	1	16	3.381	1.3311	1.3311	0.02058	0.1427	3.0050	2.6580
01/26/94	3	24.00	75.20	1	23	3.383	1.3319	1.3319	0.02108	0.1461	3.6008	2.6820
02/02/94	4	24.00	75.20	1	30	3.387	1.3335	1.3335	0.02142	0.1485	4.1075	2.6980
02/09/94	5	24.00	75.20	1	37	3.387	1.3335	1.3335	0.02163	0.1500	4.5616	2.7078
02/16/94	6	24.00	75.20	1	44	3.390	1.3346	1.3346	0.02191	0.1519	4.9700	2.7207
02/23/94	7	24.00	75.20	1	51	3.379	1.3303	1.3303	0.02211	0.1533	5.3682	2.7297
03/02/94	8	24.00	75.20	1	58	3.386	1.3331	1.3331	0.02243	0.1555	5.7130	2.7441
03/09/94	9	24.00	75.20	1	65	3.390	1.3346	1.3346	0.02286	0.1585	6.0407	2.7631
04/20/94	10	24.00	75.20	1	107	3.389	1.3343	1.3343	0.02369	0.1642	7.7527	2.7988
05/20/94	11	24.00	75.20	1	137	3.388	1.3339	1.3339	0.02393	0.1659	8.7751	2.8068
06/08/94	12	24.00	75.20	1	156	3.395	1.3366	1.3366	0.02426	0.1682	9.3445	2.8225
07/13/94	13	24.00	75.20	1	191	3.390	1.3346	1.3346	0.02451	0.1699	10.3550	2.8328

Figure D-9

PIMA RR (HCFC-141b)
BOARDS # 45 / 56, 58, 59, 61.

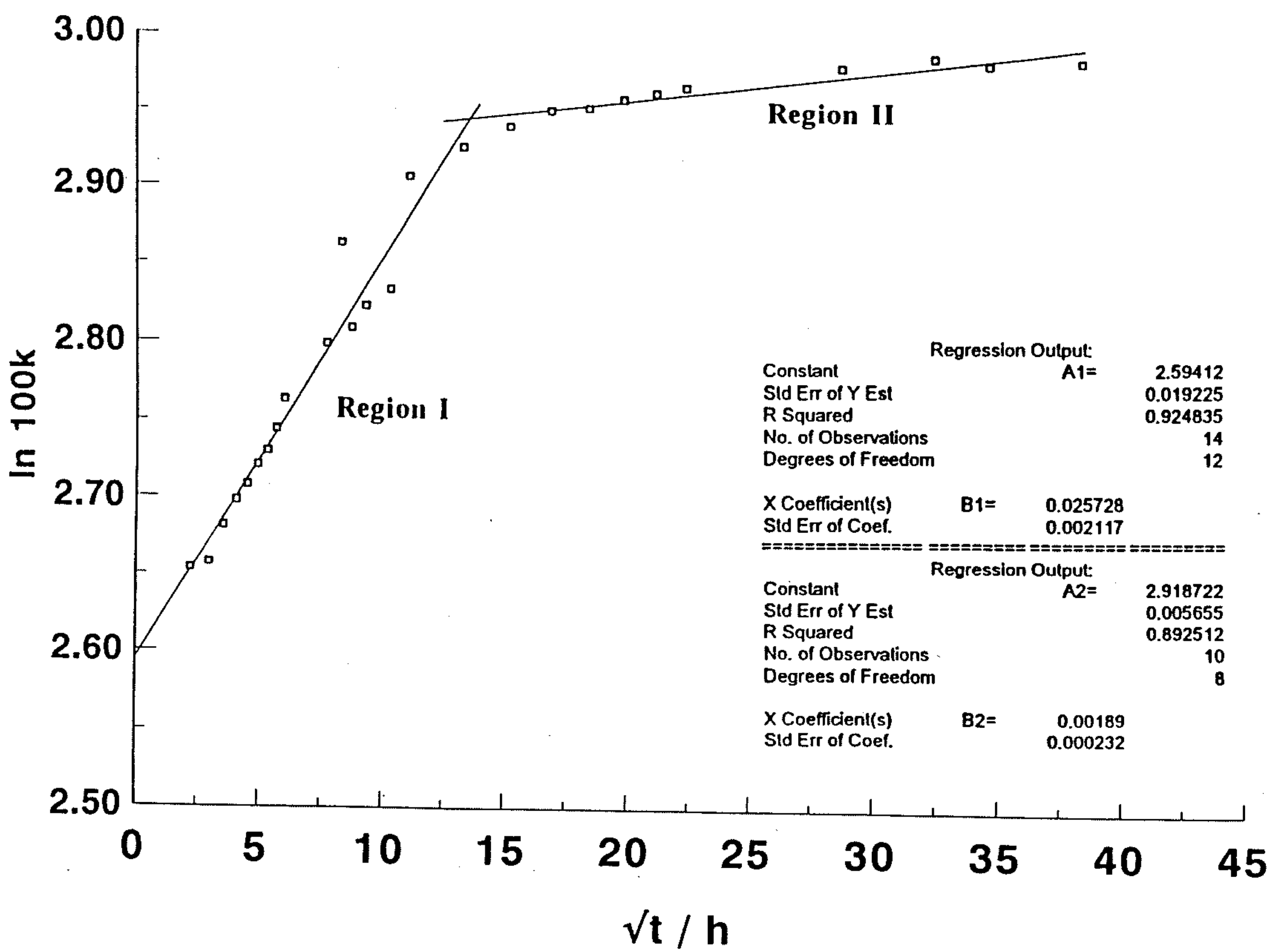


Table D-10

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant J: Specimens #48/71/72/73/74

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (In)	Avg. Aging Thickness (In)	k (w/m.K)	k (Btu.In/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/In	Y ln 100k (Btu.In/h.ft^2.F)
01/07/94	00-0000	23.00	73.40	1	4	3.393	1.3358	1.3358		0.1370	1.4972	2.6174
01/12/94	00-0000	23.00	73.40	1	9	3.399	1.3382	1.3382		0.1390	2.2418	2.6319
01/19/94	00-0000	23.00	73.40	1	16	3.414	1.3441	1.3441		0.1440	2.9760	2.6672
01/26/94	00-0000	23.00	73.40	1	23	3.449	1.3579	1.3579		0.1470	3.5319	2.6878
02/02/94	00-0000	23.00	73.40	1	30	3.452	1.3591	1.3591		0.1490	4.0302	2.7014
03/16/94	00-0000	23.00	73.40	1	72	3.432	1.3512	1.3512		0.1550	6.2799	2.7408
04/13/94	00-0000	23.00	73.40	1	100	3.432	1.3512	1.3512		0.1580	7.4009	2.7600
05/11/94	00-0000	23.00	73.40	1	128	3.432	1.3512	1.3512		0.1620	8.3732	2.7850
06/09/94	00-0000	23.00	73.40	1	157	3.432	1.3512	1.3512		0.1650	9.2733	2.8034
07/06/94	00-0000	23.00	73.40	1	184	3.434	1.3520	1.3520		0.1650	10.0333	2.8034
01/07/94	00-0000	23.00	73.40	4	4	3.805	1.4980	0.3745		0.1620	5.3403	2.7850
01/12/94	00-0000	23.00	73.40	4	9	3.810	1.5000	0.3750		0.1690	8.0000	2.8273
01/19/94	00-0000	23.00	73.40	4	16	3.861	1.5201	0.3800		0.1800	10.5258	2.8904
01/26/94	00-0000	23.00	73.40	4	23	3.861	1.5201	0.3800		0.1830	12.6200	2.9069
02/02/94	00-0000	23.00	73.40	4	30	3.861	1.5201	0.3800		0.1860	14.4130	2.9232
03/16/94	00-0000	23.00	73.40	4	72	3.871	1.5240	0.3810		0.1910	22.2708	2.9497
04/13/94	00-0000	23.00	73.40	4	100	3.881	1.5280	0.3820		0.1910	26.1788	2.9497
05/11/94	00-0000	23.00	73.40	4	128	3.820	1.5039	0.3760		0.1900	30.0909	2.9444
06/09/94	00-0000	23.00	73.40	4	157	3.823	1.5051	0.3763		0.1860	33.2996	2.9232
07/06/94	00-0000	23.00	73.40	4	184	3.886	1.5299	0.3825		0.1930	35.4650	2.9601

Figure D-10

PIMA RR (HCFC-141b)
BOARDS # 48 / 71, 72, 73, 74.

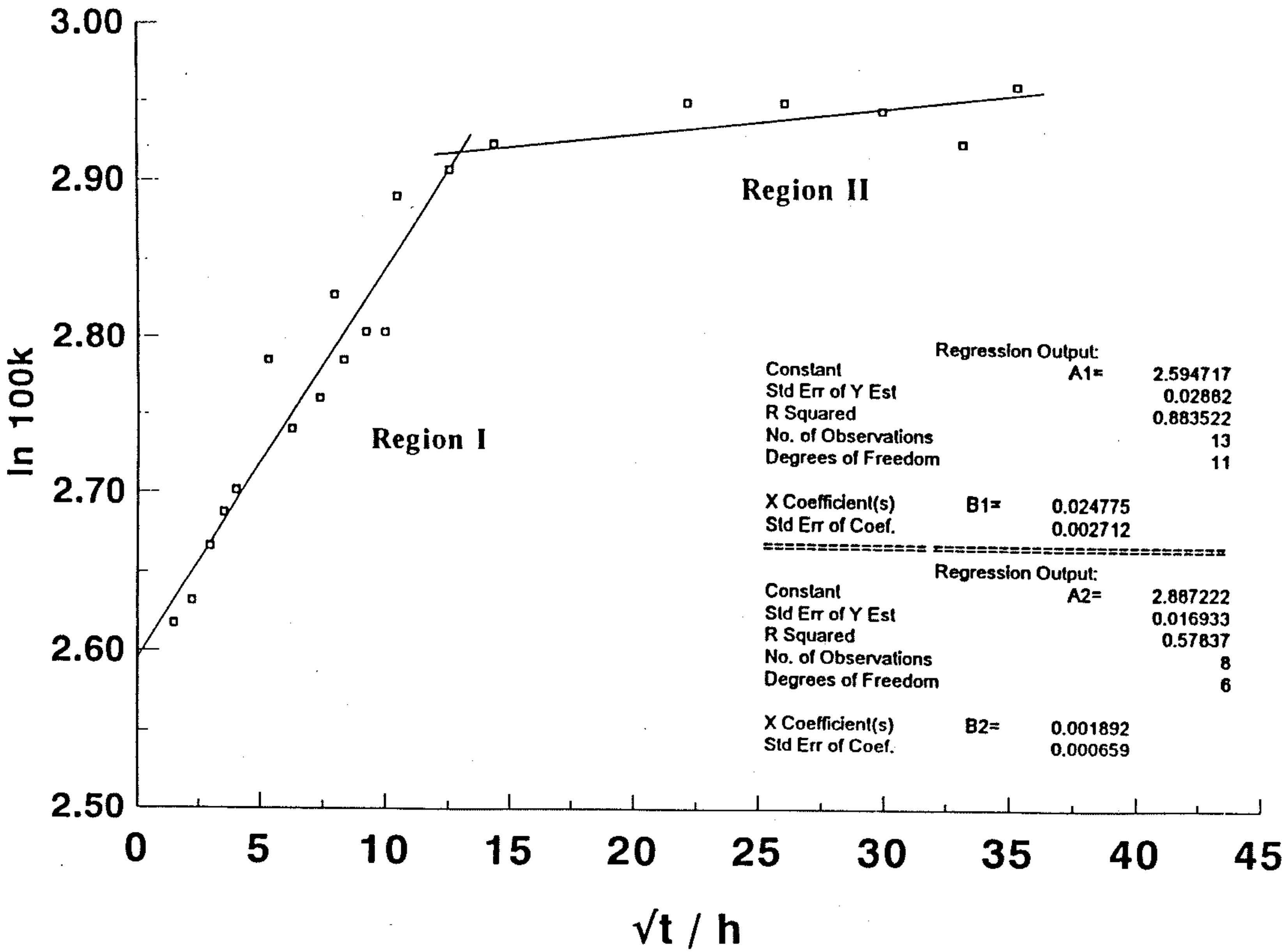


Table D-11

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant K: Specimens #83/87/88/91/92

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
01/10/94	00-0000		75.00	1	7		1.4050	1.4050		0.1560	1.8831	2.7473
01/12/94	00-0000		75.00	1	9		1.4050	1.4050		0.1560	2.1352	2.7473
01/19/94	00-0000		75.00	1	16		1.4050	1.4050		0.1580	2.8470	2.7600
01/26/94	00-0000		75.00	1	23		1.4050	1.4050		0.1610	3.4134	2.7788
02/02/94	00-0000		75.00	1	30		1.4050	1.4050		0.1630	3.8984	2.7912
03/16/94	00-0000		75.00	1	72		1.4050	1.4050		0.1680	6.0393	2.8214
04/13/94	00-0000		75.00	1	100		1.4100	1.4100		0.1720	7.0922	2.8449
05/11/94	00-0000		75.00	1	128		1.4050	1.4050		0.1730	8.0525	2.8507
06/08/94	00-0000		75.00	1	156		1.4150	1.4150		0.1750	8.8269	2.8622
07/06/94	00-0000		75.00	1	184		1.4100	1.4100		0.1760	9.6203	2.8679
01/10/94	00-0000		75.00	4	7		1.3900	0.3475		0.1700	7.6137	2.8332
01/12/94	00-0000		75.00	4	9		1.3900	0.3475		0.1740	8.6331	2.8565
01/19/94	00-0000		75.00	4	16		1.3900	0.3475		0.1790	11.5108	2.8848
01/26/94	00-0000		75.00	4	23		1.3900	0.3475		0.1820	13.8010	2.9014
02/02/94	00-0000		75.00	4	30		1.3900	0.3475		0.1840	15.7618	2.9124
03/16/94	00-0000		75.00	4	72		1.3850	0.3463		0.1870	24.5062	2.9285
04/13/94	00-0000		75.00	4	100		1.3800	0.3450		0.1880	28.9855	2.9339
05/11/94	00-0000		75.00	4	128		1.3800	0.3450		0.1880	32.7934	2.9339
06/08/94	00-0000		75.00	4	156		1.3800	0.3450		0.1890	36.2029	2.9392
07/06/94	00-0000		75.00	4	184		1.3900	0.3475		0.1900	39.0350	2.9444

Figure D-11

Figure D-11

PIMA RR (HCFC-141b)
BOARDS # 83 / 87, 88, 91, 92.

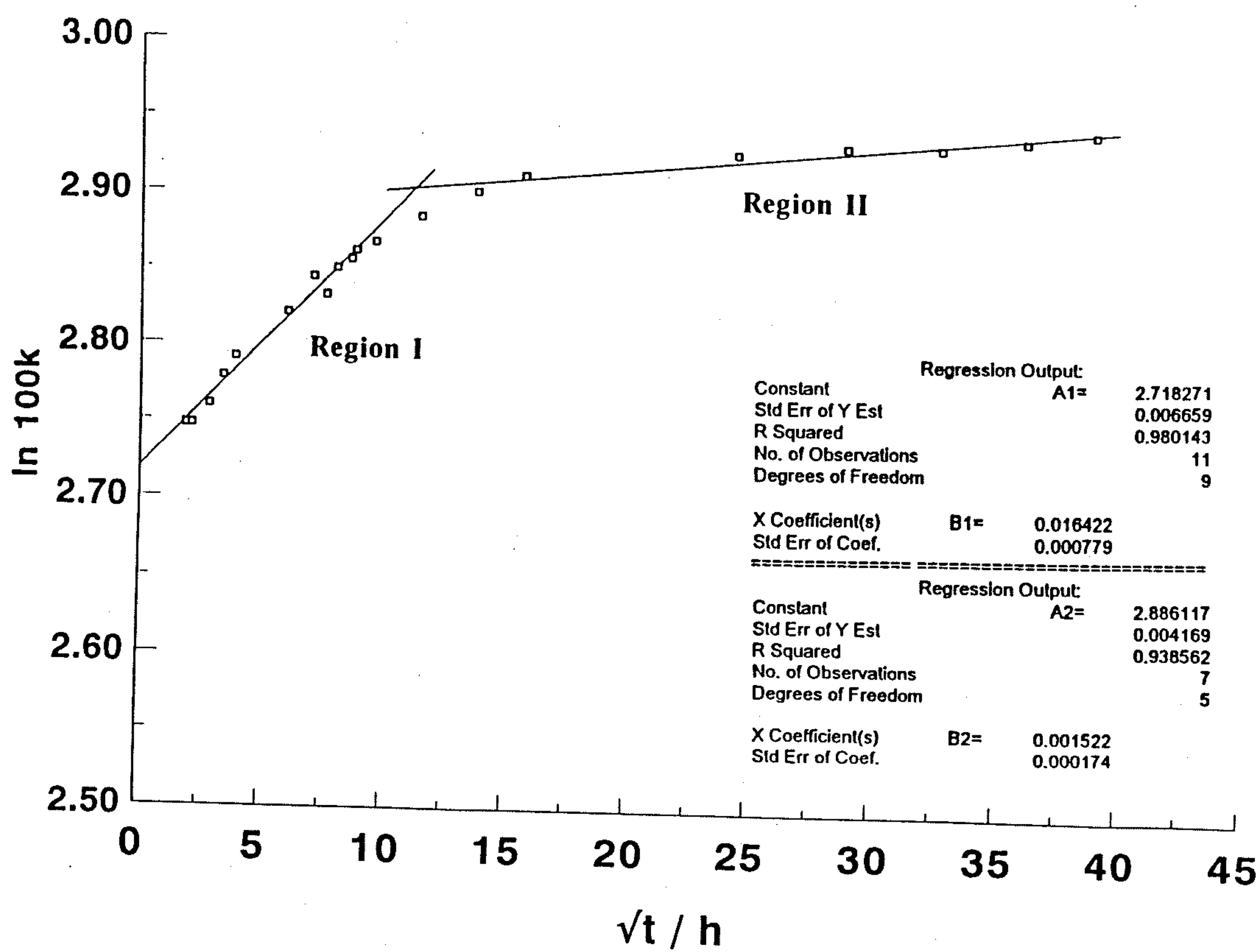


Table D-12

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant L: Specimens # 46/63/64/65/66

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
01/10/94	K940110A	23.92	75.06	1	7	3.426	1.3488	1.3488	0.02158	0.1496	1.9615	2.7056
01/10/94	K940110B	23.95	75.11	4	7	3.848	1.5150	0.3787	0.02382	0.1652	6.9857	2.8043
01/12/94	K940112A	23.94	75.09	1	9	3.426	1.3488	1.3488	0.02177	0.1509	2.2242	2.7143
01/12/94	K940112B	23.93	75.07	4	9	3.854	1.5173	0.3793	0.02433	0.1687	7.9087	2.8255
01/21/94	K940121A	23.91	75.04	1	18	3.432	1.3512	1.3512	0.02227	0.1544	3.1399	2.7367
01/21/94	K940121B	23.92	75.06	4	18	3.826	1.5063	0.3766	0.02529	0.1753	11.2664	2.8642
01/26/94	K940126A	23.94	75.09	1	23	3.434	1.3520	1.3520	0.02254	0.1562	3.5473	2.7488
01/26/94	K940126B	23.95	75.11	4	23	3.864	1.5213	0.3803	0.02600	0.1803	12.6102	2.8920
02/02/94	K940202A	23.92	75.06	1	30	3.434	1.3520	1.3520	0.02281	0.1581	4.0513	2.7609
02/02/94	K940202B	23.94	75.09	4	30	3.866	1.5220	0.3805	0.02632	0.1825	14.3944	2.9040
03/16/94	K940316A	23.96	75.13	1	72	3.426	1.3488	1.3488	0.02392	0.1658	6.2909	2.8083
03/16/94	K940316B	23.95	75.11	4	72	3.846	1.5142	0.3785	0.02715	0.1882	22.4156	2.9350
04/13/94	K940413A	23.93	75.07	1	100	3.430	1.3504	1.3504	0.02454	0.1701	7.4052	2.8338
04/13/94	K940413B	23.96	75.13	4	100	3.876	1.5260	0.3815	0.02762	0.1915	26.2126	2.9521
05/11/94	K940511A	23.91	75.04	1	128	3.430	1.3504	1.3504	0.02513	0.1743	8.3781	2.8579
05/11/94	K940511B	23.95	75.11	4	128	3.864	1.5213	0.3803	0.02789	0.1933	29.7483	2.9619
06/08/94	K940608A	23.91	75.04	1	156	3.426	1.3488	1.3488	0.02542	0.1762	9.2600	2.8691
06/08/94	K940608B	23.93	75.07	4	156	3.862	1.5205	0.3801	0.02795	0.1938	32.8582	2.9642
07/06/94	K940706A	23.90	75.02	1	184	3.436	1.3528	1.3528	0.02568	0.1780	10.0274	2.8793
07/06/94	K940706B	23.91	75.04	4	184	3.852	1.5165	0.3791	0.02802	0.1942	35.7780	2.9665

Figure D-12

PIMA RR (HCFC-141b)
BOARDS # 46 / 63, 64, 65, 66.

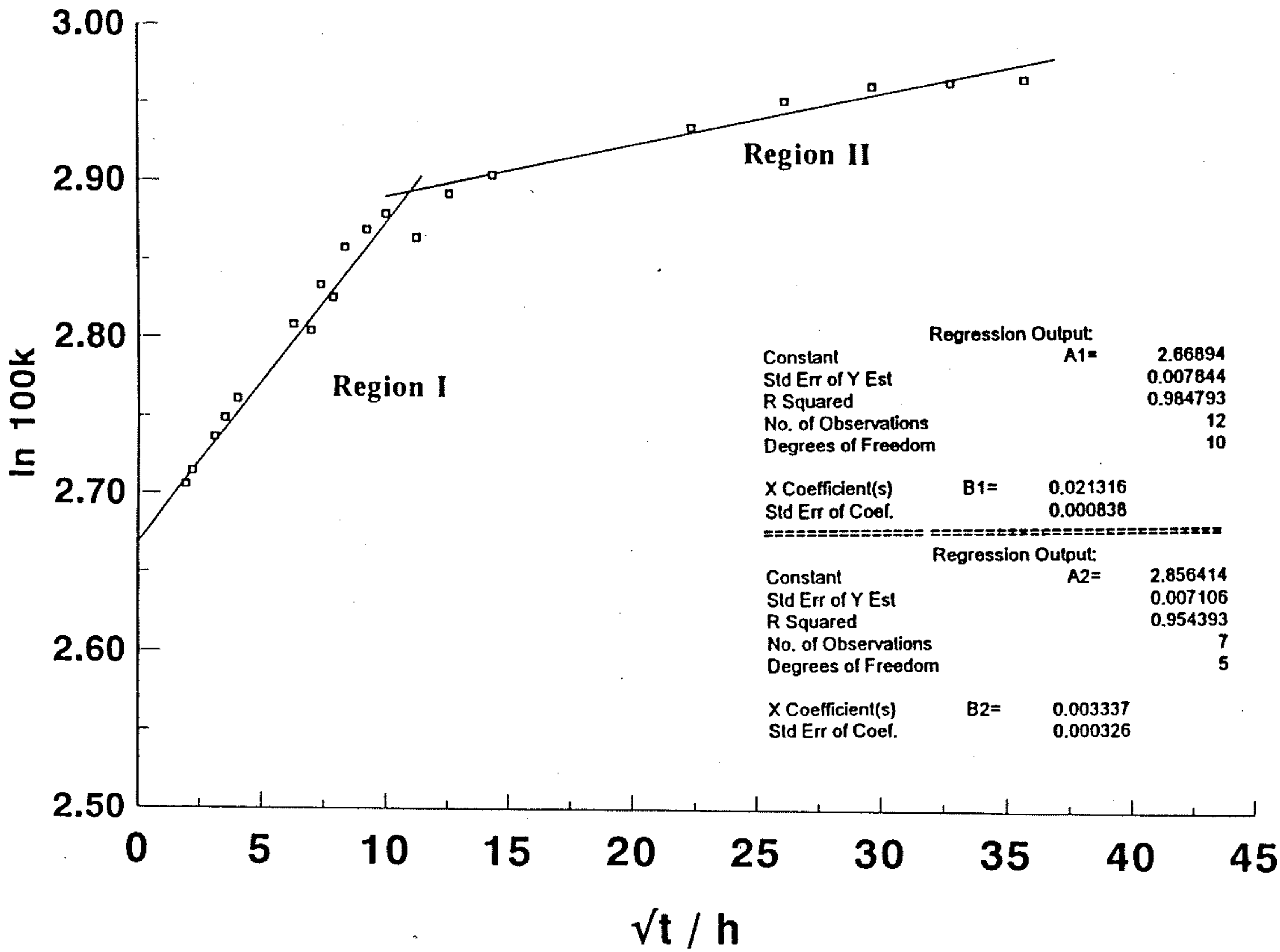


Table D-13

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant M: Specimens # 7/34/35/36/37

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
12/01/93	93-1193	23.90	75.02	1	2	3.303	1.3004	1.3004	0.01900	0.1317	1.0875	2.5781
12/10/93	93-1206	23.90	75.02	1	11	3.335	1.3130	1.3130	0.01997	0.1385	2.5260	2.6279
12/11/93	93-1207	23.90	75.02	1	12	3.310	1.3031	1.3031	0.01991	0.1380	2.6583	2.6249
12/18/93	93-1226	23.95	75.11	1	19	3.340	1.3150	1.3150	0.02029	0.1407	3.3149	2.6438
12/22/93	93-1246	24.00	75.20	1	23	3.335	1.3130	1.3130	0.02033	0.1409	3.6526	2.6458
12/29/93	93-1263	24.00	75.20	1	30	3.345	1.3169	1.3169	0.02036	0.1412	4.1591	2.6473
01/05/94	94-1011	23.93	75.07	1	37	3.334	1.3126	1.3126	0.02085	0.1446	4.6341	2.6711
01/12/94	94-1046	23.91	75.04	1	44	3.334	1.3126	1.3126	0.02119	0.1469	5.0535	2.6872
01/19/94	94-1060	23.91	75.04	1	51	3.335	1.3130	1.3130	0.02151	0.1491	5.4390	2.7022
01/26/94	94-1093	23.91	75.04	1	58	3.339	1.3146	1.3146	0.02191	0.1519	5.7934	2.7207
02/02/94	94-1131	23.91	75.04	1	65	3.332	1.3118	1.3118	0.02204	0.1528	6.1459	2.7266
02/16/94	94-1187	23.91	75.04	1	79	3.333	1.3122	1.3122	0.02308	0.1600	6.7735	2.7727
03/16/94	94-1294	23.91	75.04	1	107	3.333	1.3122	1.3122	0.02322	0.1610	7.8830	2.7787
04/13/94	94-1381	23.91	75.04	1	135	3.345	1.3169	1.3169	0.02398	0.1663	8.8228	2.8109
05/11/94	94-1464	23.91	75.04	1	163	3.338	1.3142	1.3142	0.02429	0.1684	9.7150	2.8238
06/08/94	94-1541	23.91	75.04	1	191	3.337	1.3138	1.3138	0.02489	0.1726	10.5195	2.8482
12/10/93	93-1204	23.90	75.02	4	11	3.298	1.2984	0.3246	0.02516	0.1744	10.2174	2.8590
12/18/93	93-1229	23.90	75.02	4	19	3.297	1.2980	0.3245	0.02575	0.1785	13.4323	2.8821
12/22/93	93-1247	24.00	75.20	4	23	3.294	1.2969	0.3242	0.02639	0.1830	14.7922	2.9067
12/29/93	93-1264	24.00	75.20	4	30	3.297	1.2980	0.3245	0.02677	0.1856	16.8786	2.9210
01/05/94	94-1010	24.00	75.20	4	37	3.292	1.2961	0.3240	0.02679	0.1857	18.7730	2.9217
01/12/94	94-1047	23.91	75.04	4	44	3.292	1.2961	0.3240	0.02701	0.1873	20.4720	2.9299
01/19/94	94-1059	23.91	75.04	4	51	3.289	1.2949	0.3237	0.02719	0.1885	22.0605	2.9366
01/26/94	94-1094	23.91	75.04	4	58	3.292	1.2961	0.3240	0.02732	0.1894	23.5043	2.9413
02/02/94	94-1132	23.91	75.04	4	65	3.290	1.2953	0.3238	0.02734	0.1895	24.8974	2.9421
02/16/94	94-1188	23.91	75.04	4	79	3.292	1.2961	0.3240	0.02822	0.1956	27.4314	2.9737
03/16/94	94-1295	23.91	75.04	4	107	3.290	1.2953	0.3238	0.02788	0.1933	31.9440	2.9616
04/13/94	94-1382	23.91	75.04	4	135	3.305	1.3012	0.3253	0.02816	0.1952	35.7182	2.9716
05/11/94	94-1465	23.91	75.04	4	163	3.297	1.2980	0.3245	0.02840	0.1969	39.3431	2.9801
06/08/94	94-1542	23.91	75.04	4	191	3.293	1.2965	0.3241	0.02877	0.1995	42.6401	2.9930

Figure 'D-13'

PIMA RR (HCFC-141b)
BOARDS # 7/34, 35, 36, 37.

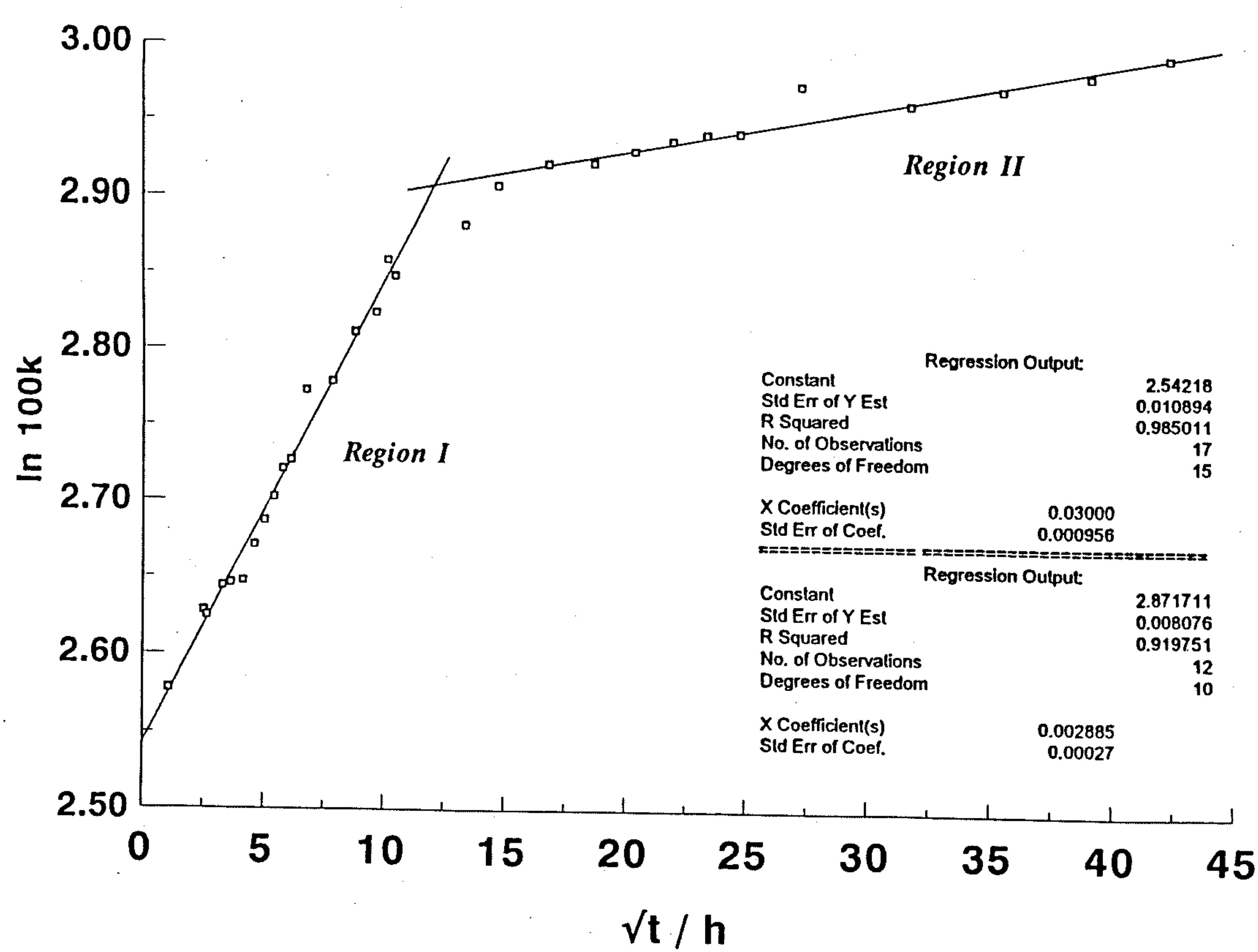


Table D-14

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant M: Specimens # 50/ 55,57,60,80

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
01/06/94	94-1014	23.91	75.04	1	3	3.401	1.3390	1.3390	0.02099	0.1455	1.2936	2.6778
01/13/94	94-1048	23.91	75.04	1	10	3.406	1.3409	1.3409	0.02145	0.1487	2.3582	2.6994
01/19/94	94-1057	23.91	75.04	1	16	3.400	1.3386	1.3386	0.02159	0.1497	2.9882	2.7059
01/26/94	94-1091	24.00	75.20	1	23	3.395	1.3366	1.3366	0.02204	0.1528	3.5880	2.7266
02/02/94	94-1128	23.91	75.04	1	30	3.404	1.3402	1.3402	0.02226	0.1543	4.0870	2.7365
02/09/94	94-1160	23.91	75.04	1	37	3.404	1.3402	1.3402	0.02279	0.1580	4.5388	2.7600
02/16/94	94-1184	23.91	75.04	1	44	3.401	1.3390	1.3390	0.02327	0.1613	4.9540	2.7809
02/23/94	94-1216	23.91	75.04	1	51	3.406	1.3409	1.3409	0.02380	0.1650	5.3257	2.8034
03/02/94	94-1241	23.91	75.04	1	58	3.406	1.3409	1.3409	0.02329	0.1615	5.6794	2.7817
03/09/94	94-1273	23.91	75.04	1	65	3.409	1.3421	1.3421	0.02337	0.1620	6.0071	2.7852
03/16/94	94-1292	23.91	75.04	1	72	3.407	1.3413	1.3413	0.02336	0.1620	6.3260	2.7847
04/13/94	94-1378	23.91	75.04	1	100	3.413	1.3437	1.3437	0.02415	0.1674	7.4421	2.8180
05/11/94	94-1462	23.91	75.04	1	128	3.411	1.3429	1.3429	0.02444	0.1694	8.4247	2.8299
06/08/94	94-1538	23.91	75.04	1	156	3.411	1.3429	1.3429	0.02508	0.1739	9.3007	2.8558
07/06/94	94-1643	23.91	75.04	1	184	3.413	1.3437	1.3437	0.02513	0.1742	10.0950	2.8578
01/06/94	94-1012	23.92	75.06	4	3	3.499	1.3776	0.3444	0.02321	0.1609	5.0293	2.7783
01/13/94	94-1049	23.91	75.04	4	10	3.507	1.3807	0.3452	0.02552	0.1769	9.1613	2.8732
01/19/94	94-1058	23.91	75.04	4	16	3.503	1.3791	0.3448	0.02622	0.1818	11.6015	2.9002
01/27/94	94-1110	23.91	75.04	4	24	3.505	1.3799	0.3450	0.02678	0.1857	14.2008	2.9214
02/02/94	94-1130	23.91	75.04	4	30	3.505	1.3799	0.3450	0.02692	0.1866	15.8769	2.9266
02/09/94	94-1161	23.91	75.04	4	37	3.508	1.3811	0.3453	0.02739	0.1899	17.6171	2.9439
02/16/94	94-1186	23.91	75.04	4	44	3.506	1.3803	0.3451	0.02796	0.1938	19.2224	2.9645
02/23/94	94-1217	23.91	75.04	4	51	3.520	1.3858	0.3465	0.02836	0.1966	20.6128	2.9787
03/02/94	94-1242	23.91	75.04	4	58	3.503	1.3791	0.3448	0.02774	0.1923	22.0886	2.9566
03/09/94	94-1274	23.91	75.04	4	65	3.508	1.3811	0.3453	0.02781	0.1928	23.3502	2.9591
03/16/94	94-1293	23.91	75.04	4	72	3.504	1.3795	0.3449	0.02777	0.1925	24.6034	2.9577
04/13/94	94-1379	23.91	75.04	4	100	3.516	1.3843	0.3461	0.02815	0.1952	28.8965	2.9713
05/11/94	94-1463	23.91	75.04	4	128	3.507	1.3807	0.3452	0.02827	0.1960	32.7765	2.9755
06/08/94	94-1540	23.91	75.04	4	156	3.509	1.3815	0.3454	0.02875	0.1993	36.1637	2.9923
07/06/94	94-1644	23.91	75.04	4	184	3.512	1.3827	0.3457	0.02876	0.1994	39.2417	2.9927

Figure 'D-14'

PIMA RR (HCFC-141b)
BOARDS # 50/55, 57, 60, 80.

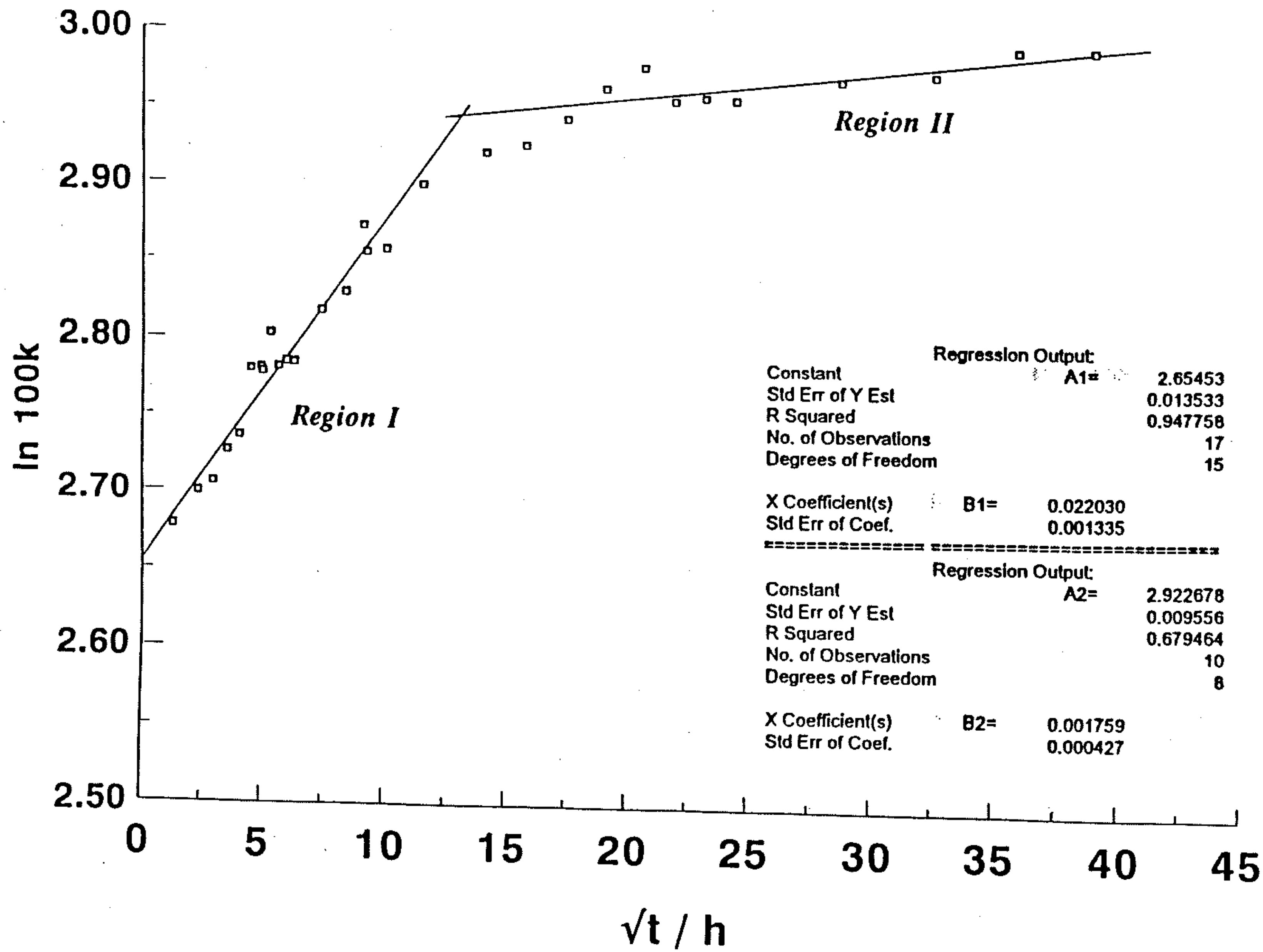


Table D-15

PIMA/SPI/ORNL CRADA
Interlaboratory Comparison HCFC141b
Participant M: Specimens # 82/85,86,89,90

Date	Test	Temp. (C)	Temp. (F)	Slices In Test	Age (days)	Test Thickness (cm)	Test Thickness (in)	Avg. Aging Thickness (in)	k (w/m.K)	k (Btu.in/h.ft^2.F)	X (Sqrt Time)/Aging Th. (Sqr Days)/in	Y ln 100k (Btu.in/h.ft^2.F)
01/11/94	94-1037	23.87	74.97	1	8	3.581	1.4098	1.4098	0.02003	0.1389	2.0062	2.6309
01/24/94	94-1088	23.88	74.98	1	21	3.562	1.4024	1.4024	0.02116	0.1467	3.2678	2.6858
01/27/94	94-1100	23.88	74.98	1	24	3.560	1.4016	1.4016	0.02092	0.1450	3.4953	2.6744
02/02/94	94-1129	23.87	74.97	1	30	3.580	1.4094	1.4094	0.02106	0.1460	3.8861	2.6811
03/16/94	94-1291	23.87	74.97	1	72	3.591	1.4138	1.4138	0.02235	0.1550	6.0018	2.7405
04/14/94	94-1390	23.81	74.86	1	101	3.594	1.4150	1.4150	0.02347	0.1627	7.1026	2.7894
05/11/94	94-1461	23.87	74.97	1	128	3.598	1.4165	1.4165	0.02342	0.1624	7.9869	2.7873
06/08/94	94-1539	23.83	74.89	1	156	3.603	1.4185	1.4185	0.02445	0.1695	8.8050	2.8303
07/06/94	94-1642	23.83	74.89	1	184	3.605	1.4193	1.4193	0.02443	0.1694	9.5573	2.8295
01/11/94	94-1039	23.87	74.97	4	8	3.499	1.3776	0.3444	0.02603	0.1805	8.2129	2.8930
01/24/94	94-1083	23.88	74.98	4	21	3.472	1.3669	0.3417	0.02748	0.1905	13.4098	2.9472
01/27/94	94-1103	23.88	74.98	4	24	3.477	1.3689	0.3422	0.02708	0.1877	14.3151	2.9325
02/02/94	94-1133	23.87	74.97	4	30	3.491	1.3744	0.3436	0.02727	0.1891	15.9406	2.9395
03/16/94	94-1288	23.87	74.97	4	72	3.515	1.3839	0.3460	0.02807	0.1946	24.5264	2.9684
04/14/94	94-1386	23.81	74.86	4	101	3.503	1.3791	0.3448	0.02787	0.1932	29.1484	2.9613
05/11/94	94-1466	23.87	74.97	4	128	3.512	1.3827	0.3457	0.02833	0.1964	32.7299	2.9776
06/08/94	94-1543	23.83	74.89	4	156	3.506	1.3803	0.3451	0.02831	0.1963	36.1946	2.9769
07/06/94	94-1648	23.82	74.88	4	184	3.474	1.3677	0.3419	0.02824	0.1958	39.6710	2.9744

Figure D-15

PIMA RR (HCFC-141b)
BOARDS # 82 / 85, 88, 89, 90.