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PROBLEMS IN PREDICTING THE THERMAL PROPERTIES OF FACED POLYURETHANE FOAMS

by Mark Bomberg

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SOMMAIRE

Les propriétés thermiques des mousses de polyuréthane évoluent dans le temps en fonction des conditions écologiques, du mode de mise en place et de la présence d'une membrane de revêtement dès leur production. L'effet d'un revêtement sur le rendement thermique d'un composite va d'une légère amélioration de sa stabilité dimensionnelle à une réduction sensible du taux de perte de résistance thermique.

Il conviendrait de traiter les polyuréthanes à revêtement comme des composites pour la mise à l'essai, suivant une méthode qui reproduit le plus fidèlement possible les conditions réelles. Il conviendrait aussi de déterminer la composition des gaz dans les cellules comme mesure du procédé de vieillissement. Une telle détermination après exposition à différentes conditions ambiantes permettrait une corrélation approximative entre les résultats obtenus en laboratoire et le rendement réel.

L'auteur présente des renseignements de base sur l'effet des facteurs écologiques et des simulations de vieillissement sur la résistance thermique des polyuréthanes et propose un point de départ pour l'étude de la méthode d'essai.



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Problems in Predicting the Thermal Properties of Faced Polyurethane Foams

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ABSTRACT: The thermal properties of polyurethane foams vary during service life depending on environmental conditions, the manner in which they are enclosed in the building partition, or if covered with facing membranes during their production. The effect of facing materials on thermal performance of the composites may range from a slight improvement of dimensional stability to a substantial reduction in the rate of loss of thermal resistance. Faced polyurethanes should be tested as composite materials, using a method that simulates performance conditions as closely as possible. Such a test method should include determination of cell gas composition as a measure of the aging process. Determination of cell gas composition after a period of exposure to different service conditions would allow an approximate correlation of laboratory results with *in situ* performance. The paper gives background information on effects of environmental factors and aging simulations on thermal resistance of polyurethanes and provides a starting point for discussion of the testing method.

KEY WORDS: thermal resistance, thermal conductivity, polyurethane foams, faced polyurethanes, urethanes, aging, design, thermal properties, thermal insulation

During the production process the cells of polyurethanes are filled with a captive blowing agent, for example, refrigerant gas. The refrigerant gas and carbon dioxide (if present) diffuse outward; the surrounding air diffuses inwards, into the material cells. These diffusion processes occur at different rates; the slowest is the refrigerant gas. At the final stage all pores and cells of the material are filled with a mixture of air, water vapor, and a small fraction of blowing agent. The rate of diffusion depends on the chemical and physical structure of the polyurethane foam and the conditions of exposure.

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The complete diffusion process can require several months to more than a hundred years.

Increasing the rate of gas diffusion is the most effective way to simulate, in the laboratory, the so-called aged² thermal properties of polyurethane foams. The basis of its effectiveness is that higher temperatures accelerate all the diffusion processes that occur in polyurethane foams.

In one of the laboratory conditioning procedures a specimen 25 mm thick is cut from a sample and its thermal resistance measured. It is then placed in an oven at a given temperature, either 60 or 100°C, for 28 days and the thermal resistance measured again. There may be some correlation between thermal resistance after 28 days' exposure at 100°C and the estimated *in situ* value for some materials, but the correlation is poorly established.

The method in current use in Canada for laboratory conditioning is based on dry heat conditioning for 28 days at the prescribed temperature of 100°C. There are many arguments for and against the 100°C exposure.

Arguments in favor are:

1. It causes a fast reduction of thermal resistance.
2. It helps to distinguish between "satisfactory" and "unsatisfactory" foams.

Against this procedure, one could state the following:

1. It is rather improbable that polyurethane would be exposed to such conditions. Such an exposure might introduce some changes in the "chemistry" of the material.
2. Environmental stresses occurring during actual service may be entirely different from those in the hot, dry conditioning exposure.
3. At a temperature of 100°C, the compressive strength, particularly in the direction perpendicular to the rise of the polyurethane [*I*]³ may fall below the level of stress introduced by the air pressure difference, causing rupture of some cells. This would probably not occur at lower temperatures.
4. 100°C exceeds the limit for "continuous use surface temperature" [2] indicated in materials standards; for example, Canadian Government Specification Board (CGSB) Standard 51-GP-21M specifies the limit at 80°C.

The procedure of 28 days' aging at 100°C has been used for many years in the industry and performs the role of quality control. Specimens exposed to high-temperature conditioning are usually 25 mm thick with dimensions suitable for inserting into the apparatus for measuring thermal properties, that is, 300 by 300 mm. The present method of accelerated laboratory conditioning is, however, not suitable for faced polyurethanes, as materials faced with impermeable gas barriers cannot be sliced to 25 mm thickness. It

²Reduction in thermal resistance of polyurethane foams is termed "aging."

³The italic numbers in brackets refer to the list of references appended to this paper.

may be possible to obtain different thermal resistances for the same material when conditioning and testing specimens with different geometries.

If this procedure were applied to the composites, the facing would have to be removed and the specimen cut to 25-mm thickness, or a set of new requirements relating geometry and exposure time would have to be introduced.

It is advisable, therefore, to consider whether a new method can be established to test the composite product instead of its core component. Such a test method should not only distinguish between "satisfactory" and "unsatisfactory" foams but also incorporate a procedure whereby the thermal resistance of laboratory-conditioned specimens could be compared with *in situ* performance.

Background Information on the Loss of Thermal Resistance of Polyurethanes

Most refrigerant gases have a lower thermal conductivity than air. For example, with air rated as 100 percent at 0°C, carbon dioxide and trichlorofluoromethane would have thermal conductivities of 60.7 and 34.5 percent, respectively [3]. The thermal conductivity of a mixture of any two gases can be estimated from the sum of the products of the thermal conductivities of the components and their molar concentrations. This simple equation was discussed by Skochdopole [4] and shown to deviate by not more than 10 percent from the actual thermal conductivity of a gaseous mixture.

If one knows the composition of cell gas, that is, the mole fractions of air and other gases, such as captive blowing gas, and also the initial apparent thermal conductivity⁴ of the material, one can estimate the thermal conductivity of the conditioned specimen. Nadeau et al [3] developed an equation and showed that it agreed well with results of measurements carried out in a guarded hot-plate apparatus on polyurethanes with small uniform pores. Data calculated for foams with a coarse irregular cell structure (and particularly those with distorted cells) were not in close agreement with the measured thermal conductivities.

Composition of a cell gas can be estimated from knowledge of conditions of exposure, material composition, and pore structure, and the specimen geometry if the appropriate diffusion coefficients are known.

Norton [5] used the diffusion equation to determine the ratio between blowing gas and air in the pores and change of thermal conductivity due to

⁴ Apparent thermal conductivity is the correct term to describe thermal properties of fibrous and cellular thermal insulations (λ app). Since it is generally accepted, the word "apparent" is omitted for brevity.

gas diffusion. He discussed four characteristic regions relating thermal conductivity to duration of exposure:

1. Initially air diffuses inward; carbon dioxide, if present, diffuses outward. This results in a rise in thermal conductivity.
2. A plateau occurs when the diffusion of air and carbon dioxide is approaching equilibrium and outward diffusion of refrigerant gas has not caused a significant increase in thermal conductivity.
3. Outward diffusion of the refrigerant gas causes a slow rise in thermal conductivity. This process continues until all the blowing gas has diffused from the cells.
4. The final stage occurs when an equilibrium mixture with a small percentage of the blowing gas is present inside the cells.

Ball et al [6] identified the effect of average pore size (cell diameter) of polyurethanes on the change in thermal conductivity. Tests by Harding and James [7] showed that density changes in the range 29 to 37 kg/m³ did not significantly affect thermal conductivity and that the thermal conductivity perpendicular to the rise direction of molded foam varied by as much as 20 percent from that in the parallel orientation. The study also showed that the closed-cell content was of primary importance.

The increase in thermal conductivity due to one-dimensional diffusion⁵ in polyurethanes with 100 percent uniform closed cells was modeled by Dixon et al [8]. The increase in thermal conductivity of R11 blown polyurethane is thoroughly discussed in the literature [9] where Norton's theoretical equations were used to predict the thermal conductivity with reasonable accuracy provided the diffusion coefficients of the particular foam were known as well as the initial temperature and partial pressures of the gases in the cells.

Effect of Environmental Factors on Thermal Properties of Polyurethanes

The following factors related to environmental conditions should be considered when discussing thermal properties of polyurethanes during their service life:

1. mean temperature,
2. mean relative humidity of the ambient air,
3. changes of mean temperature during service life,
4. temperature gradient across the material,

⁵In nonstationary diffusion processes the permeability coefficient may differ from the diffusion coefficient if the specific gas content (sometimes called "solubility," that is, the gas content in the solid polymer and cell space of the material) is variable. These terms are often used interchangeably in the literature on polyurethanes and cause confusion.

5. differences in humidities of the ambient air on each side of the layer or temperature gradient causing moisture vapor transmission through the material,

6. presence of water on the hot surface of the material in combination with temperature gradients.

Studies of some of these environmental factors have been reported in the literature. Mean temperature was found to be of major significance for the diffusion process, and laboratory conditioning at elevated temperatures became standard practice in conditioning polyurethane foams. Exposure at an elevated temperature may cause additional stresses which may cause some cell walls to break. Thermal conductivity measurements carried out after 28 days' conditioning at 100°C (given in Table 1) identify a few materials where cell walls have broken. It is not known to what extent these cells would be broken in a long time exposure at a lower temperature.

The effect of temperature cycling has been studied by Bettanini et al [10,11]. Figure 1, taken from this research, illustrates the test conditions and the results obtained. From these one can conclude that temperature cycling contributes nothing more to the aging process than does steady exposure at the same level of mean temperature.

In a review of the factors affecting thermal conductivity in service, Paljak [12] presented the data shown in Fig. 2. Two specimens were exposed to a steady temperature of 20°C. One specimen was kept in an environment with 25 percent relative humidity; the other was cycled between 25 and 90 percent relative humidity. The latter specimen showed greater increase in thermal conductivity than the one kept in a dry atmosphere.

The effect of moisture vapor transmission on the thermal effectiveness of polyurethanes was studied by Levy [13]. Figure 3 shows the thermal conductivity measured on a wall erected between a freezer having an air temperature of -29°C and a warm chamber with an air temperature of 38°C and approximately 100 percent relative humidity. Thermocouples inserted in the center of polyurethane insulation 10 cm thick indicated the average apparent thermal conductivity for the two layers with various mean temperatures. A parallel series of tests with the same material, but protected with hot-applied asphalt as a vapor barrier, showed an increase in thermal conductivity of only a few percentage points on the warm side; the average value did not change more than 2 to 3 percent during the 16 months of tests.

Figure 3 also illustrates the effect of moisture accumulation on the thermal properties of polyurethane foams. Thermal resistance of the material exposed to the higher temperature was lower than that of the material exposed to lower temperatures, even though the same driving force of water vapor transmission was applied in all tests.

Another study concerning thermal conductivity of polyurethanes at low

TABLE 1—Results of thermal conductivity tests on polyurethane specimens, 25 mm thick. Materials were 1 to 10 weeks old when received. The specimens were stored in a 50% RH 22°C room for a few days prior to the first test. Different products are denoted by letters from a to r and various specimens by numbers.

Product and Specimen Designation	Thermal Conductivity, W/m · K		
	Initial	Conditioned for 28 Days at 100°C	Conditioned at 22°C, 50% RH
a1	0.0179	0.0219	0.0235 after 17 months
a2	0.0185	0.0224	...
a3	0.0198
b1	0.0199	0.0333	...
c1	0.0202	0.0228	0.0234 after 15 months
d1	0.0228	0.0250	...
e1	0.0198
e2	0.0190
f1	0.0189	0.0239	...
f2	0.0189	...	0.0232 after 28 months
g1	0.0180	0.0206	0.0224 after 12 months
g2	0.0190
h1	0.0196
i1	0.0213
j1	0.0185
k1	0.0192	0.0262	...
k2	0.0202	0.0275	...
l1	0.0195	0.0226	0.0252 after 12 months
l2	0.0195	...	0.0224 after 12 months
m1	0.0202	0.0335	...
m2	0.0196	0.0335	...
m3	0.0209	...	0.0286 after 145 months
n1	0.0208
n2	0.0183	...	0.0238 after 18 months 0.0261 after 48 months 0.0291 after 130 months
p1	0.0183
r1	0.0202

temperatures performed by Bigolaro et al [14] examined thermal conductivity of a polyurethane foam as a function of temperature and temperature gradient in the range between 110 and 300 K for a few weeks. Thermal conductivity was found stable within 0.2 percent except where there was a peak in the curve at, for example, 236 K, where it was within 0.4 percent.

References [10–16] contain discussions of environmental factors in relation to thermal conductivity measured during a particular environmental exposure. Hilado [1] discusses the effect of environmental factors in terms of dimensional stability of the material. Following the study of three types of aging environments—cold, dry atmosphere; hot and dry; and hot, humid conditioning—he came to the following conclusion: “A combination

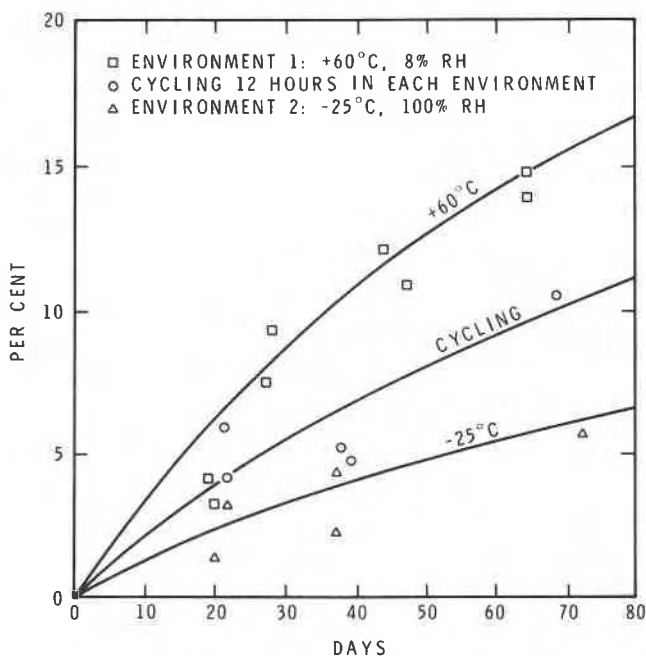


FIG. 1—Percent variation of thermal conductivity of a 63-mm-thick specimen, with density 29 kg/m^3 , cut from a board stock. Initial value of thermal conductivity $\lambda = 0.020 \text{ W/m} \cdot \text{K}$. (from Bettanini et al [10]. Reprinted with permission of the authors.)

of high temperature and humidity is more likely to threaten the integrity and performance of the material than dry heat alone or low temperature."

Accelerated Laboratory Conditioning

Refrigerant gas trichloromonofluoromethane (Cl_3CF) has a diffusion coefficient of $2.25 \times 10^{-9} \text{ cm}^2/\text{s}$ at 25°C . Rating it as 100 percent one obtains 533 percent at a temperature of 60°C and 1644 percent at 90°C [4]. Exposing the material to elevated temperatures will accelerate the diffusion and shorten the testing time.

Figure 4 shows the results of tests on specimens 25 mm thick that were conditioned at 100°C and thermal conductivity measurements made at 0°C . Two curves were calculated and compared with the results of the measurements; the only difference between the two sets of calculations is in the diffusion constants employed [9].

The difference between Curves A and B in Fig. 4 makes it necessary to modify the diffusion coefficients used in Norton's equation. There may be several reasons for these differences, for example, the effect of a particular

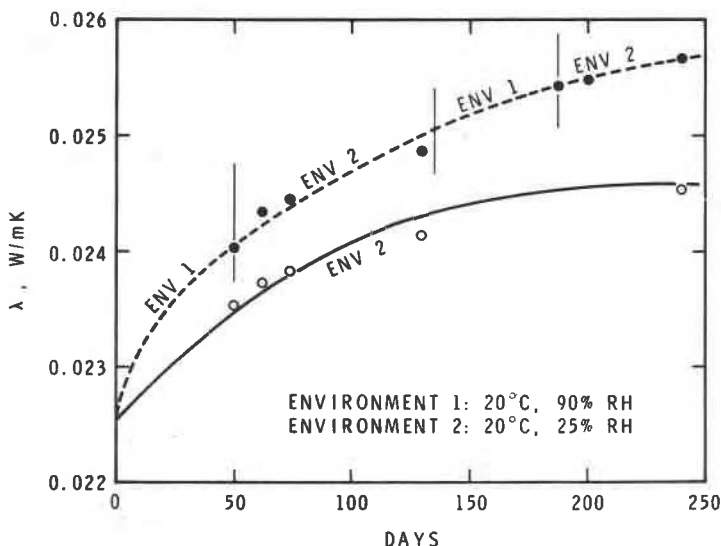


FIG. 2—Thermal conductivity of polyurethane (30 kg/m^3) as a function of time. Aging in steady environment compared with variation in relative air humidity. (After Paljak [12]. Reprinted with permission of the author.)

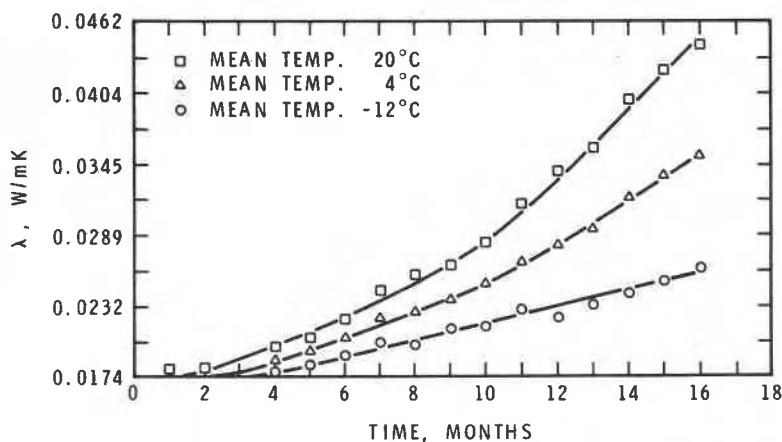


FIG. 3—Thermal conductivity of "wet" urethane foam versus time. Basis: unprotected installation, no vapor barrier. Moisture vapor driving force: 44 mm Hg. (After Levy [13]. Reprinted with permission of Technomic Publishing Company, Westport, Conn.)

structure of the tested specimen, a different temperature dependence of the diffusion coefficients, or cell-breaking due to exposure at elevated temperatures. Patten and Skochdopole [15] showed that crushing cell walls increased thermal conductivity of the polyurethane.

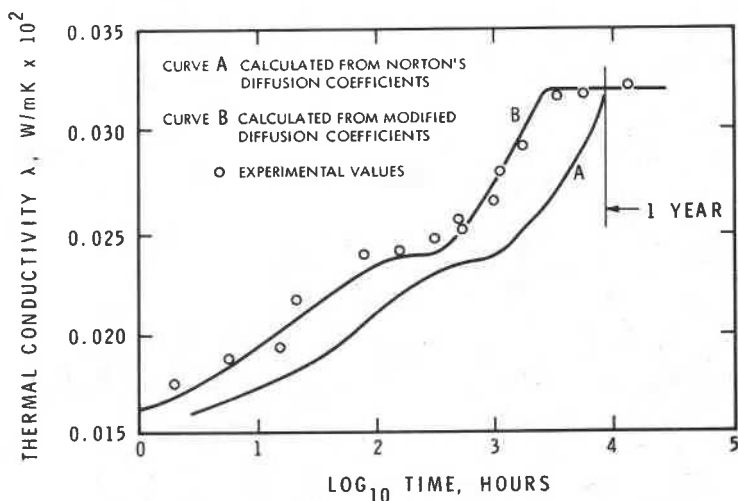


FIG. 4—Comparison of calculated and measured thermal conductivity curves for specimens conditioned at 100°C and measured at 0°C . (samples 25 mm thick) [9].

The effect of elevated temperature is evident in some of the values shown in Table 1, which gives some results from tests carried out at the Division of Building Research, National Research Council of Canada, during the past 12 years. Because many of the products have been reformulated, these results are not representative of the polyurethane foams produced at present. They are also applicable to 25-mm specimens only. Some general conclusions, however, can be drawn from the information in Table 1, as follows.

Several specimens were stored in an air-conditioned room with temperature about 22°C and 50 percent relative humidity. Thermal conductivity measurements gave an average thermal conductivity $\lambda = 0.022 \text{ W/m} \cdot \text{K}$ after 1 year, $\lambda = 0.024 \text{ W/m} \cdot \text{K}$ after about 2 years, and (about a 9 percent increase), $\lambda = 0.029 \text{ W/m} \cdot \text{K}$ after 11 to 12 years' storage (an increase of about 32 percent when compared with measurements after 1 year's storage).

The thermal conductivity of specimens of the same materials conditioned for 28 days at 100°C can be compared with these results. Several measurements fall in the range of $\lambda = 0.021$ to $0.024 \text{ W/m} \cdot \text{K}$ (Products *a*, *c*, *e*, *g* and *l*); some results show thermal conductivity in a range between 0.025 and $0.028 \text{ W/m} \cdot \text{K}$ (Products *d* and *k*); Products *b* and *m* appear to reach the maximum possible reduction in thermal resistance with $\lambda = 0.033 \text{ W/m} \cdot \text{K}$, indicating a final equilibrium. In the case of Product *m* both specimens conditioned for 28 days at 100°C indicate possible cell breakage since a third specimen, which had been in storage for more than 12 years, showed an increase in thermal conductivity that was only 68 percent of the increase during the accelerated laboratory conditioning.

There is a significant scatter between the test results in Table 1, which may be partly due to uncertainty as to the age of the specimens. The thermal conductivity of Specimen *a3* before exposure differs by about 9 percent from the average for Specimens *a1* and *a2*. The same scatter is also present for the conditioned specimens. This means that either the thermal resistance of a selected specimen should be noted as a function of time and exposure conditions, or statistically significant numbers of specimens must be tested to provide an average thermal resistance for that material.

A similar variability of the results was shown by Kuester [17] when comparing several curves of thermal conductivity versus exposure time determined by different researchers.

A Possible Method to Accelerate Laboratory Conditioning of Faced Polyurethanes

A variety of facing materials is used on polyurethanes but only a few act as effective barriers to gas diffusion and affect the thermal performance of materials.

Other materials provide resistance to moisture intrusion and improve dimensional stability and perhaps, to a lesser extent, thermal resistance. These products will not perform in the same way as fully protected urethanes nor as unprotected, unfaced urethanes. They must be tested as composites and the test method should be as close as possible to conditions in actual use. The test method should fulfill two functions:

1. Indicate thermal performance of a foam system under severe environmental stresses.
2. Facilitate a correlation of *in situ* thermal performance with thermal performance of the laboratory-conditioned material.

The test method under consideration consists of the following:

1. Either a climatic exposure of a sample from which specimens are taken for thermal resistance tests, or intermittent thermal resistance measurements on a conditioned sample.
2. Determination of the average cell gas composition two or three times during laboratory conditioning, preferably at the time of thermal resistance testing. The cell gas composition should be determined as the mean for the whole thickness of the specimen (see the Appendix).
3. Measurement of thermal resistance at the beginning of the exposure, and after a specified time, for example, 28 or 90 days.

Conditioning of faced polyurethanes must be done on larger slabs; appropriate specimens for testing could be cut from the conditioned materials. Immediately after cutting the specimens to be used for thermal conductivity

measurements, the edges of the specimens must be sealed, for example, by applying self-adhesive aluminium foil. If metal facing is used on the surfaces, a slot will have to be cut in the facing material at the junction between main and guard heaters immediately before testing. This slot should be covered with an impermeable plastic tape to prevent air from entering the specimen.

Specimen preparation forms a very important part of the test method as is illustrated in Table 2, which shows test results on two fresh and two conditioned specimens of the same sample. The specimens had a nominal thickness of 25 mm and were covered with aluminum facing on both sides. Both fresh (unaged) and aged specimens showed differences in thermal resistance when the cut edges were left exposed for some time.

Requirements regarding sampling techniques to determine cell gas content and for thermal conductivity testing are not included in this paper. They must be specified in detail to ensure reproducibility of the conditioning procedure.

Two testing methods seem to be worthy of consideration:

1. Placement of the material specimen between two steady environments, for example, $70^{\circ} \pm 2^{\circ}\text{C}$ and 97 ± 3 percent relative humidity [ASTM Test for Response of Rigid Cellular Plastics to Thermal and Humid Aging (D 2126-75), Procedure C] and $23^{\circ} \pm 2^{\circ}\text{C}$ and 50 ± 5 percent relative humidity (room conditions) for three months (90 days).
2. Exposing materials to successively larger temperature gradients with an intermittent thermal resistance determination as proposed by Sherman [18] at the Thermal Insulation Conference.

Although Method 1 utilizes almost maximum continuous temperature and humidity conditions to which the materials could be exposed, the mean temperature is low, 46°C , and it is quite possible that thermal resistance may change very quickly and remain constant.

Method 2 gives information on a larger span of the diffusion process as well as a record of temperature on the hot side when a significant foam cell degradation was indicated by the inability to return to the reference thermal performance level. It would appear that a 600-mm heat flowmeter apparatus could easily be adapted for such testing and that Method 2 should be carefully examined by manufacturers and testing laboratories.

Concluding Remarks

Norton's theory allows a prediction of thermal resistance as a function of time from a knowledge of specimen dimensions, initial cell gas composition, conditions of exposure, and diffusion coefficients appropriate for a particular cell structure of polyurethane foam. It is concerned with the change in composition of the gaseous mixture in the cell and expresses thermal

TABLE 2—*Thermal resistance determined on polyurethane foam (Code No. 314-59) after applying different conditioning procedures. (All specimens had sealed edges during the test.)*

Aging and Conditions Prior to Testing	Specimen Dimensions, mm	Thermal Resistance per Unit Thickness		Notes
		$K \cdot m^2/W$	$ft^2F/h/Btu$	
None, 2 days in laboratory	450 by 450 by 27.1	54.9	7.92	edges sealed immediately
None, 2 days in laboratory	200 by 200 by 26.6	52.9	7.81	edges sealed after 2 days
100°C, 28 days (1200 by 1200 mm)	200 by 200 by 21.9	46.9	6.76	cut prior to testing
100°C, 28 days	200 by 200 by 18.6	45.6	6.57	edges exposed during conditioning

resistance as a linear function of the mole fraction of refrigerant in the air [4,5,11]. Such an equation is characteristic of a particular type of polyurethane foam.

To determine the fraction of refrigerant gas in the pore air, Fick's law of diffusion has been used [5,16]. Several research studies on movement of moisture in a capillary system [19] indicated that Fick's law is applicable when the appropriate diffusion coefficient corresponds to the combined liquid and vapor transport⁶ processes. A reason for this might be that using the "modified diffusion constants" produced good agreement with the experimental values (Fig. 4), while the diffusivity coefficient originally determined (Curve A) produced a shift in the time scale. One way to establish diffusivity coefficients would be to determine a few curves of concentration distribution after various periods of exposure to the same environmental conditions as the exposure for which thermal resistance is sought. Because it is impossible to determine the homogeneity of a block of material and because of an additional resistance to diffusion created by skins or facing, it is not practicable to use modified diffusion coefficients to estimate the diffusion coefficients of faced materials.

It is proposed that the laboratory testing of thermal properties be extended to include the determination of cell gas content. From this, a formula could be established which would be valid for each product tested in the laboratory. (The thermal resistance would be expressed as a linear function of the mole fraction of the refrigerant gas.) This formula could be utilized only after specimens had been exposed for a sufficient number of years to provide the gas mixture required for its determination. The polyurethane

⁶ In a capillary system exposed to a temperature gradient, movements in liquid and gaseous phases occur simultaneously and often in both directions, resulting in a change of the effective "diffusivity" coefficient.

foam specimens should be drilled across their whole thickness to determine the average cell gas composition and thus allow an estimate of the corresponding thermal resistance for selected climate and service conditions.

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APPENDIX

Analytical Report of Polyurethane Rigid Foam Board

[Analysis carried out by A. Desaulniers and R. Ironside, Analytical Section, Division of Chemistry, National Research Council of Canada (Lab. No. 16650, 19 Jan. 1978)]

The polyurethane rigid foam board was sampled by cutting "cores" through its thickness with a No. 13 cork borer. These cylinders measured about 22 mm in diameter and 23 mm in length. Their weight was about 0.220 g.

To identify the captive blowing agent, a large hypodermic syringe was filled with "cores" and the needle was inserted into an evacuated infrared gas cell through a septum; the "cores" were crushed by driving home the piston of the syringe. The infrared spectrum of the gas so obtained established that it was trichloromonofluoromethane, Cl_3CF (Freon 11).

One or two of the cylinders just described were burned in the Parr oxygen bomb in 35 atm of oxygen, the combustion products being absorbed in 10 ml of 0.2-N sodium carbonate solution. The chlorine was determined in the absorbent by titrating with N/10 silver nitrate solution after acidification with nitric acid.

The total chlorine content of three specimens tried was 8.8, 9.4 and 10.0 percent by weight.

In order to correct this total chlorine value for any chlorine compounds in the nonvoid body of the plastic, a few "cores" were ground finely in a mechanical pulverizer and the fine powder so obtained was degassed in vacuo for 30 min. Microscopic examination confirmed that no intact cells had survived the grinding process. Two specimens of this powder were then treated as described above and gave the following values: 4.6 and 4.7 percent weight/weight chlorine.

The corrected values for the chlorine in the cell volume, namely, 4.2, 4.8, and 5.4 percent weight/weight, were calculated to Cl_3CF to give *Freon 11 contents of the specimen of 5.4, 6.2, and 7.0 percent by weight.*

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