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## ***Measurements of the Rate of Gas Diffusion in Rigid Cellular Plastics***

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## RÉSUMÉ

Ce document décrit une méthode de mesure de diffusion des gaz dans les plastiques cellulaires. Un échantillon est placé dans une chambre, qui est ensuite fermée hermétiquement et mise en pression à l'aide du gaz d'essai. L'augmentation de la pression fait pénétrer une certaine quantité de gaz dans l'échantillon, ce qui réduit la pression dans la chambre. En analysant l'évolution de cette pression en fonction du temps, on détermine le coefficient de diffusion du matériau.

Les auteurs décrivent l'application de cette méthode à des échantillons provenant de la partie centrale d'un panneau de polystyrène extrudé.

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# Measurements of the Rate of Gas Diffusion in Rigid Cellular Plastics

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**ABSTRACT:** This paper describes a method for measuring gas diffusion through cellular plastics. A specimen is placed in a chamber, which is then sealed and pressurized with the test gas. The pressure increase causes some gas to enter the specimen, and this entry results in a reduction of the chamber pressure. By following the change in chamber pressure with time an appropriate transport characteristic for the material is determined.

An application of this method to a core specimen of an extruded polystyrene board is presented.

## INTRODUCTION

**G**AS-FILLED CELLULAR PLASTICS (GFCP) are usually manufactured using blowing agents, e.g., chlorofluorocarbons (CFC), that provide these products with thermal resistance higher than that of air-filled cellular plastics. During the service life of GFCP, a simultaneous inward diffusion of air components and outward diffusion of the blowing agent take place. This causes a progressive dilution of the blowing agent present in the cell-gas and results in decreased thermal efficiency, a process often referred to as aging. Thus, to predict the rate of changes in thermal performance of the GFCP one needs information on the rate of transfer for all gases which participate in the aging process.

Usually, rate of gas transfer through a material is measured on a specimen placed between two chambers with different gas pressures; the rate of gas flow and the corresponding pressure difference are then measured. These measurements [1-4] require careful sealing of the specimen to the partition

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dividing the high and low pressure chambers. Performing these types of measurements, the authors found that effective sealing around the specimen could be achieved only for cylinders with thickness larger than the diameter. Attempts to increase the diameter to thickness ratio were unsuccessful because the specimen would deflect and damage the seal when the pressure difference was applied.

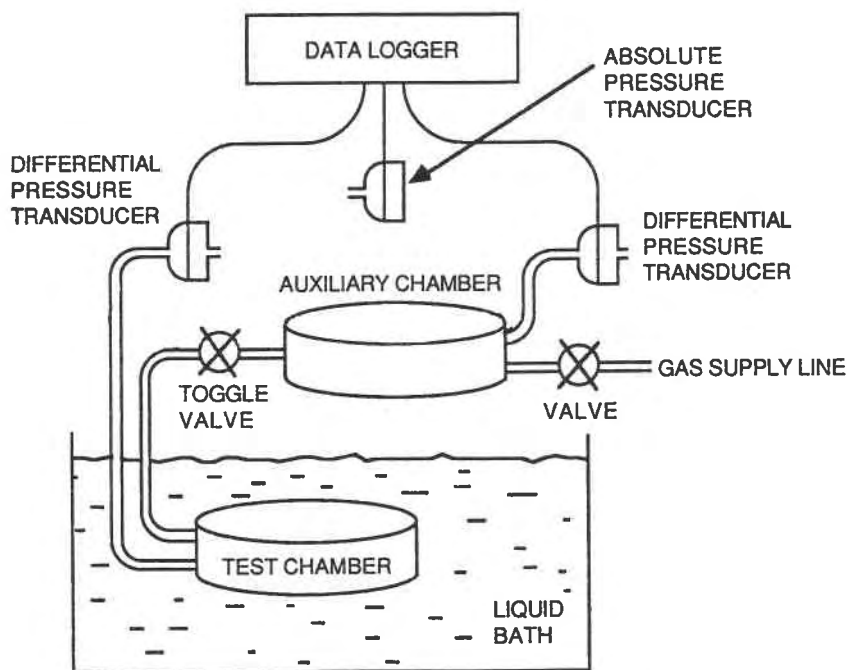
Since the main difficulty in these measurements is with the quality of the seal, any method which does not require a seal around the specimen would be worth considering. Therefore, a concept proposed in 1963 [5], in which the specimen is placed inside a sealed chamber was selected for this investigation. In this method [5], the initial gas pressure throughout the whole specimen volume had to be uniform. This condition may not be satisfied when testing CFC transfer through a CFC-blown cellular plastic. As the rate of CFC transfer is 10 to 1000 times lower than that of the air components, it takes 10 to 1000 times longer to condition the specimen with CFC than with air. Therefore, a non-uniform distribution will prevail even after lengthy conditioning of the specimen. The CFC transfer, therefore, needs to be determined from another experiment; e.g., from the measurement of thermal resistivity versus time.

The fact that the aging process of GFPC involves the transfer of two gases (or more) in and out of the specimen, leads to an additional requirement; the method must permit the analysis of a simultaneous transfer of a minimum of two gases. To perform calculations of chamber pressure versus time one must solve numerically a set of differential equations. These equations, one for each gas being transferred, describe the conservation of mass along the gas flow path, from chamber to the specimen center. Similar equations were solved by the procedure developed for an aging model [6], and therefore this procedure was used for calculations of the pressure chamber—time curves.

## METHOD

### Experimental Set-Up

A schematic of the experimental set-up is shown in Figure 1. A test chamber is immersed in a liquid bath which is equipped with a heater and a controller maintaining the required temperature within 0.02 C. The test chamber is connected, through a valve, to an auxiliary chamber, which in turn is connected through another valve to a supply of pressurized gas. Each of the chambers is connected to a differential pressure transducer with a measuring range of 0 to 133 kPa, measuring the difference between atmospheric and chamber pressures. An absolute pressure transducer, with measuring range of 0 to 133 kPa, is used to measure the atmospheric pressure. All pressure



**FIGURE 1.** Schematic representation of the set-up to measure the rate of gas transfer through cellular plastics.

transducers are connected to a PC computer used as the data acquisition system.

The test chamber is a brass cylinder with internal diameter of 153 mm, depth of 38 mm and cell wall thickness of 8 mm. The cylinder is soldered to the bottom plate. The top plate is removable and is fastened by eight bolts to the bottom plate. The sealing between the top plate and the cylinder walls is achieved by an indium gasket positioned in a groove of the top plate and melted before each assembly of the chamber. The pressure lines, of 6 mm copper tubing, are soldered to the brass chamber.

### Test Procedure

#### SPECIMEN CUTTING

Specimens are prepared in two stages. First a thin sheet approximately 2 mm thicker than the required specimen thickness is cut using a horizontal band-saw. Discs, 150 mm in diameter, are then cut from this sheet and smoothed to the required thickness with a vertical spindle surface grinder equipped with a Carborundum disc.

## SPECIMEN CONDITIONING

As previously stated, one must know the initial pressure distribution of all gases considered in the experiment. (In case of CFC, one may estimate its pressure distribution with help of the aging model calculations.) The gas pressure distributions are known either for a freshly manufactured foam or for the foam subjected to a suitable conditioning. The length of the conditioning period is approximated assuming a diffusion into a slab under condition of constant ambient gas pressure and for a Fourier number of 0.5 [7]. Thus, for the specimen with thickness,  $L$ , and an estimated effective diffusion coefficient,  $D$ , the required period,  $t$ , may be calculated as:

$$t = 0.5 \cdot L^2/D \quad (1)$$

## MEASUREMENT OF EFFECTIVE (CLOSED-CELL) VOLUME

The measurement is performed in an apparatus shown in Figure 2, which consists of two cylindrical polymethylmethacrylate (Plexiglass) chambers. The source chamber with a volume  $V_1 = 650$  ml, is connected through a valve to a supply of pressurized air, and through another valve to a sample chamber. A differential pressure transducer, with measuring range up to 13 kPa, is attached to the source chamber. The sample chamber has an inside diameter of 175 mm and volume  $V_2 = 980$  ml. The sample chamber is equipped with a valve connecting it to atmospheric pressure.

The measurement starts with the specimen in the sample chamber and both chambers at atmospheric pressure. The valve connecting the two chambers is closed and the source chamber is pressurized to about 13 kPa above atmospheric pressure. The pressure difference is recorded as  $p_1$ . The valve connecting both chambers is then opened and after pressure in both chambers has equalized, the differential pressure reading is recorded as  $p_2$ . Using the ideal gas law, the effective specimen volume,  $V_{eff}$ , is given by:

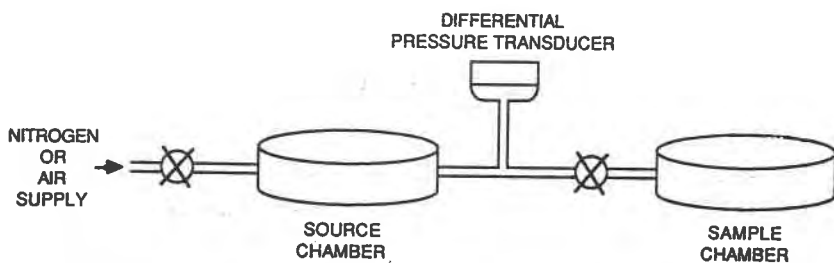


FIGURE 2. Schematic representation of the set-up to measure the effective volume of the specimen.

$$V_{eff} = V_2 - V_1 \cdot (p_1 - p_2)/p_2 \quad (2)$$

In this research, the effective volume was determined on three or four slices stacked together, and reported as the average of nine repeated measurements.

#### THICKNESS OF DESTROYED SURFACE LAYER (TDSL)

Specimen thickness is measured with a micrometer. For calculations of gas transfer, however, the specimen thickness must be corrected for the thickness of the destroyed surface layer (TDSL) [8]. This correction accounts for the fact that during specimen preparation some cells at the material surface are broken. To determine the TDSL, one measures the effective volume of the specimen using a gas displacement technique, and calculates the difference between geometric and effective specimen volume. This difference divided by the specimen surface area gives the thickness of the destroyed surface layer.

#### PRESSURE MEASUREMENTS

After the specimens are placed in the test chamber and sealed the recording of pressure in the auxiliary and test chamber is started. Now, the auxiliary chamber, which is used to impose a controlled pressure step in the test chamber, is pressurized to the required level and the valve to the test chamber is opened. This valve is closed when the pressure in both chambers is equalized (approximately 5 seconds). The output from the absolute pressure transducer which measures atmospheric pressure, and the output from the differential pressure transducer are added to give the absolute pressure in the chamber. This is done by a computer used as the data acquisition system. The chamber pressure-time curve may now be used to determine the effective diffusion coefficient.

Since only one diffusion coefficient may be determined from a single experiment, the need to accommodate simultaneous diffusion of two or more gases implies a need for performing two or more experiments and then using an iterative procedure to optimize all determined material characteristics. Such an iterative procedure to calculate diffusion coefficients for oxygen, nitrogen and CFC, comprising three steps, is presented below.

*Step 1.* A conditioned specimen is placed in the test chamber, the oxygen pressure in the chamber is increased in the manner discussed above and the decay of the chamber pressure is measured. To calculate an approximate transport coefficient for oxygen, one may either take a CFC diffusion coefficient from the literature or disregard CFC diffusion for this initial calculation. Then, assuming an oxygen diffusion coefficient one calculates a pressure decay curve and compares it with the measured one. The value of the oxygen diffusion coefficient is then adjusted proportionately to the differ-

ence between measured and calculated curves. This process is repeated until the agreement between measured and calculated chamber pressure-time curves is judged acceptable, i.e., when it gives, in a graphic display, a sufficient correspondence with measurements.

Identical operations are then performed after completing the measurement of chamber pressure-time curves for nitrogen diffusion.

*Step 2.* Using the approximate oxygen and nitrogen diffusion coefficients, under given initial (oxygen, nitrogen, CFC concentrations) and boundary conditions, the change in thermal resistivity with time is calculated with the aging model [6] and compared with the results of measurements (using a thin layer of the material). Different values of the CFC diffusion coefficient are tried, until a sufficient correspondence with the measurement is obtained.

*Step 3.* Using the above determined CFC diffusion coefficient, the chamber pressure-time curves are calculated again, as in step 1, and steps are repeated until the final selection of the effective diffusion coefficient for oxygen and nitrogen is made.

### Use of the Relative Pressure

Use of the relative pressure is convenient when comparing tests performed on the same material but under different conditions. The dimensionless relative pressure is defined as:

$$\bar{P} = (P - P_f)/(P_i - P_f) \quad (3)$$

where  $\bar{P}$  is the relative pressure,  $P$  is the actual pressure which changes from the initial value of  $P_i$  to the final value at equilibrium,  $P_f$ . To use the relative pressure, however, one must either measure or estimate the initial and final pressure values. If a constant diffusion coefficient is assumed, one may use Equations (4) and (5) to calculate the initial and final pressures.

The process of gas transfer from the chamber to the specimen comprises two stages. When pressure is increased in the test chamber, the gas enters holes and crevices on the specimen surface, some of which are induced during the sample preparation. Only after a certain period, when the pressure reaches some initial level,  $P_i$ , the second stage of gas transfer process begins and gas starts to diffuse into the closed-cell volume of the specimen. At this moment the process becomes independent of the open cells and cracks adjacent to the material surfaces.

To determine the pressure separating these two stages,  $P_i$ , one must know the quantity of gas injected to the test chamber. This information comes from the use of a calibrated volume, called here an auxiliary chamber. Thus, the initial pressure level is determined from Equation (4):



$$P_i = P_p + (P_{ax} - P_p) \frac{V_{ax}}{V_{fr} + V_{ax}} \quad (4)$$

where  $P_p$  is the equilibrium pressure before the introduction of the gas from the auxiliary chamber,  $P_{ax}$  is the pressure level in the auxiliary chamber,  $V_{ax}$  is the volume of the auxiliary chamber,  $V_{fr}$  is the free volume of the test chamber, i.e., the difference between the chamber volume,  $V_{ch}$ , and the effective volume of the specimen,\*  $V_e$ .

For an ideal gas, the final equilibrium pressure is:

$$P_f = \frac{P_i \cdot V_{fr} + P_p \cdot V_e \cdot (1 - d_r)}{V_{fr} + V_e \cdot (1 - d_r)} \quad (5)$$

where  $d_r$  is the ratio between densities of the foam and the solid polymer.

Having defined the initial and final pressure levels, the actual pressure may be presented in the form of a relative pressure, Equation (3). Moreover, if the use of Equations (4) and (5) is permissible and the final equilibrium pressure is estimated, the measurements may be ended before reaching the final equilibrium conditions.

## APPLICATION OF THE METHOD

### Materials

All measurements discussed in this paper were performed on the middle part (core) of an extruded polystyrene with average density of 35.5 kg/m<sup>3</sup>. Measurements of gas transfers were made on three discs with mean thickness of 8.64 mm and diameter of 150.2 mm. Measurements of thermal resistivity were made on 5.2 mm thick, 300 mm square specimens. The core layer of the extruded polystyrene is assumed to be homogeneous enough to disregard differences in structure of 5 and 9 mm thick layers.

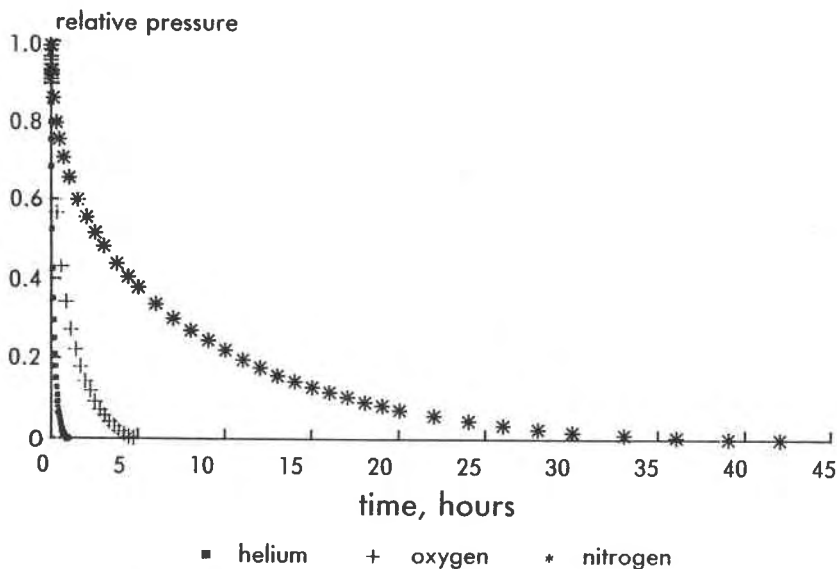
This material was also characterized with the values of the initial thermal resistivity and the mass extinction coefficient. The latter property, measured with an infrared spectrophotometer by the Division of Physics NRCC, gave an average mass extinction coefficient of 46 m<sup>2</sup>/kg.

### Results

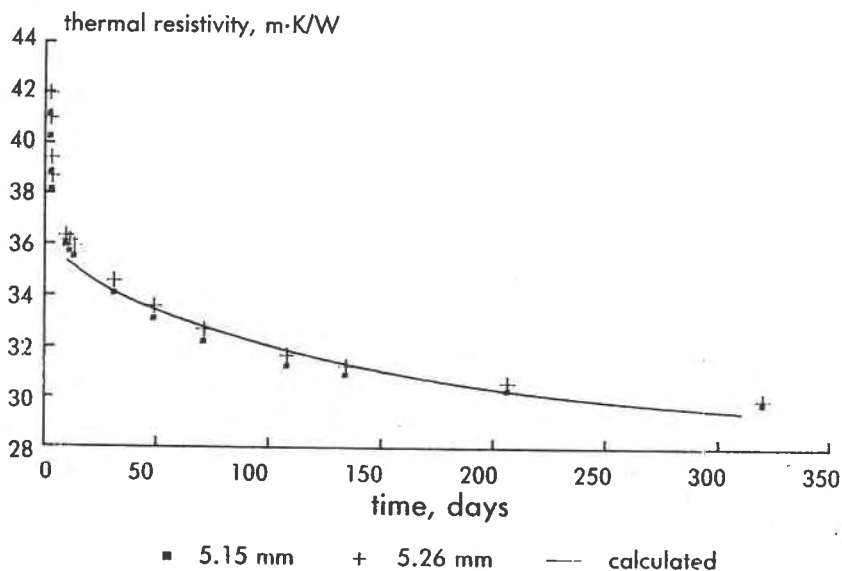
Results of the measurements of helium, oxygen and nitrogen diffusion are shown in Figure 3. Results of the measurements of thermal resistivity versus time, that were performed on 5 mm thick specimens, are shown in Figure 4.

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\* For semi-flexible and flexible foams, a correction must be introduced for specimen compression under increased pressure.



**FIGURE 3.** Relative chamber pressure curves for helium, oxygen and nitrogen diffusion measurements.



**FIGURE 4.** Aging curve for core layers of extruded polystyrene; the continuous curve was calculated using model of aging [6] and the experimentally determined oxygen, nitrogen diffusion coefficients. Selected CFC-12 diffusion coefficient is  $1 \times 10^{-13} \text{ m}^2/\text{s}$ .

Table 1. Measurements of the thickness of the destroyed surface layer from the difference between geometrical,  $V_g$ , and effective,  $V_e$ , volumes and the standard deviation in  $V_e$  measurements on core of the extruded polystyrene board.

Edge Seal	Geom. Thick. (mm)	Difference ( $V_g - V_e$ ) (ml)	Standard Deviation (%)	Destroyed Layer (mm)
no	8.80	19.1	0.76	0.18
yes	7.86	17.2	2.08	0.16
yes	8.57	19.3	1.48	0.18
yes	8.64	21.4	1.97	0.20

Results of the measurements of the thickness of destroyed surface layer (TDSL) are given in Table 1.

The best correspondence with measured aging curve, in step 2 listed above, was obtained with a CFC-12 diffusion coefficient of  $1 \times 10^{-13}$  m<sup>2</sup>/s, see Figure 4. Using this CFC-12 diffusion coefficient, the measured mass extinction coefficient of 46 m<sup>2</sup>/kg and assumed initial pressure distribution of CFC-12 (estimated in such a manner that the measured initial thermal resistivity of 48 m·K/W would be obtained), calculations of the chamber pressure were performed for nitrogen and oxygen. It was found that the best correspondence to the chamber pressure curves, in step 3, was given by an effective diffusion coefficient  $4.6 \times 10^{-11}$  m<sup>2</sup>/s for oxygen, cf Figure 5, and  $6.5 \times 10^{-12}$  m<sup>2</sup>/s for nitrogen, cf Figure 6.

The uncertainties in the optimized values for the diffusion coefficients are discussed below. However, to check the reliability of the values arrived at, two independent series of measurements were performed on the same material as follows.

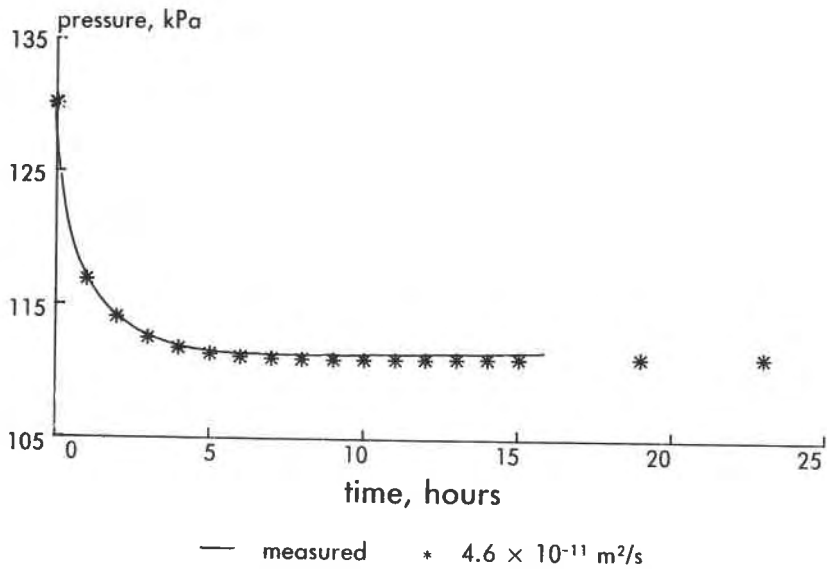
In one series, changes in nitrogen pressure in the chamber were introduced as five successive step changes, as shown in Figure 7. The figure also shows calculated changes in pressure for which the previously determined diffusion coefficients were used. The agreement between the measured and calculated pressure changes is good.

In the second series, one side of the specimen was coated with an epoxy resin to simulate one-sided nitrogen diffusion. Figure 8 compares the experimental data with those calculated using the previously determined diffusion coefficient. Once again the agreement is sound.

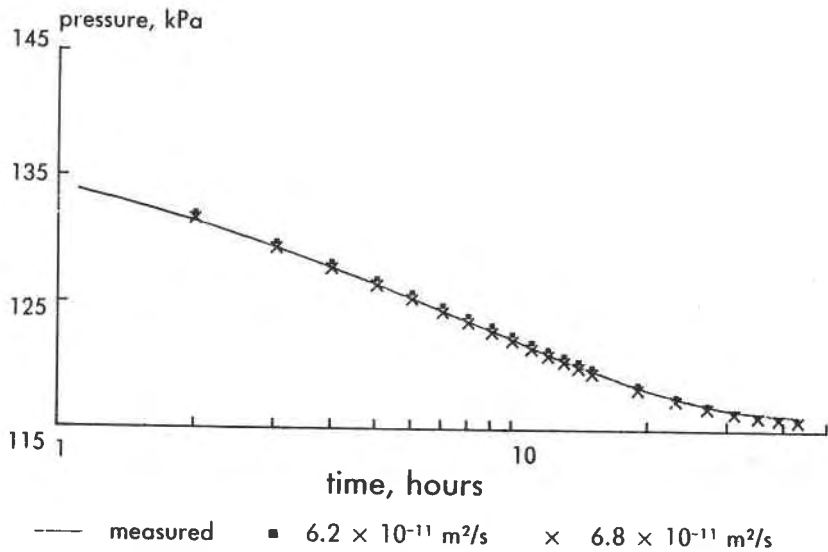
#### UNCERTAINTY OF THE DIFFUSION COEFFICIENT DETERMINATION

There are two sources of uncertainty in effective diffusion coefficient:

1. Uncertainty in pressure and effective volume measurements



**FIGURE 5.** Measured and calculated oxygen chamber pressure curves. Oxygen diffusion coefficient of  $4.6 \times 10^{-11} \text{ m}^2/\text{s}$  was used in the calculation.



**FIGURE 6.** Measured and calculated nitrogen chamber pressure curves. Nitrogen diffusion coefficients  $6.2 \times 10^{-12} \text{ m}^2/\text{s}$  and  $6.8 \times 10^{-12} \text{ m}^2/\text{s}$  were used.

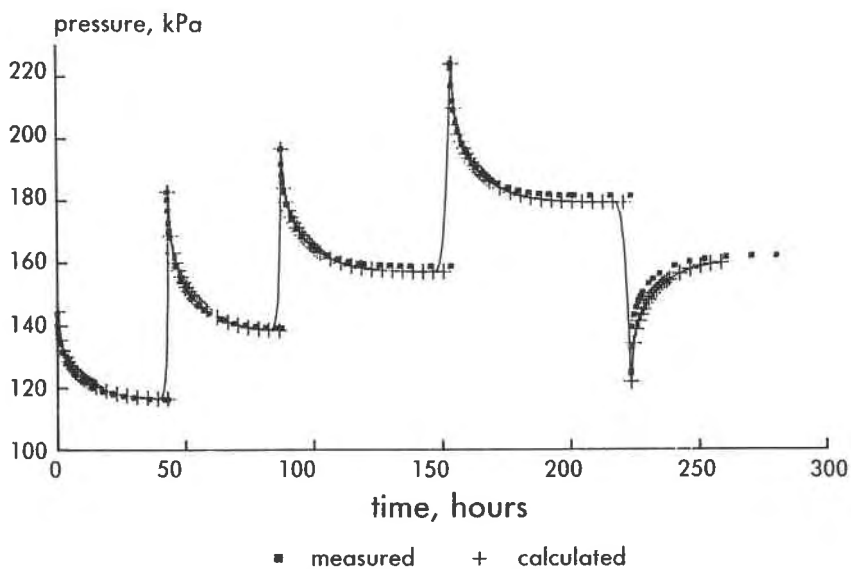


FIGURE 7. A series of step changes in nitrogen pressure.

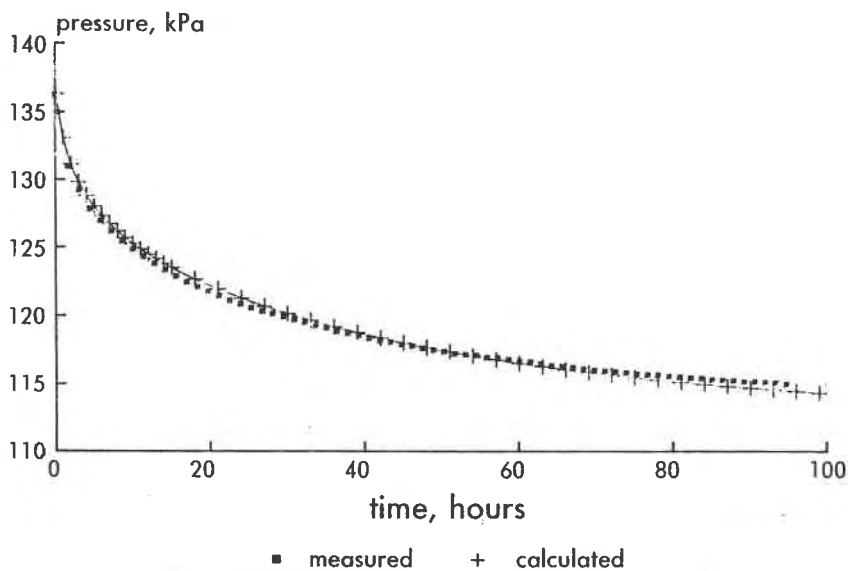


FIGURE 8. Nitrogen diffusion from one side.

2. Uncertainty in selection of the diffusion coefficient that gives best fit to the measured chamber pressure-time curves

The first source of uncertainty has much smaller contribution to the total uncertainty than the second source. The uncertainties in physical measurements are as follows:

1. The standard deviation of series of the thickness measurements performed on a specimen with carefully ground surface was 0.03 mm. Normally, 7 measurements are performed on a specimen with 5 mm thickness. Assuming normal distribution of errors ( $t$ -distribution) and probability level of 95%, one may calculate a two-sided confidence interval. For 7 measurements at 95% probability  $t$ -factor of 1.9 may be found [9], and the percentage uncertainty of thickness measurement becomes  $(1.9 \times 0.03)/(5 \times 7) = 0.0043$  or 0.43%.
2. Percentage uncertainty of diameter measurement, even though only three readings are taken is much smaller because it relates to a large measured value. For the same standard deviation of 0.03 mm and 95% probability level but three measurements,  $t$ -factor is 2.35 and the uncertainty is  $(2.35 \times 0.03)/(150 \times 3) = 0.0003$  or 0.03%.
3. The differential pressure transducer is calibrated with a transfer standard with an accuracy 0.08 percent.
4. The uncertainty of the data acquisition system is estimated 0.1 percent.
5. A 25 mm thick aluminum disc was precision machined and the geometric volume of 431.98 cm<sup>3</sup> was determined. Using the technique described above, the effective volume was determined as 432.61 cm<sup>3</sup>, i.e., within 0.2% of the geometric volume. The standard deviation of these measurements was 1.8 cm<sup>3</sup>. Normally, 9 measurements are made on a specimen with approximately 400 cm<sup>3</sup> effective volume. For 9 measurements and for 95% probability level,  $t$ -factor is 1.83 and the uncertainty of effective volume is  $(1.83 \times 1.8)/(400 \times 9) = 0.0027$  or 0.27%.

The second source of uncertainty is the sensitivity of the curve fit used for selecting diffusion coefficient. The importance of this uncertainty source is displayed in Figure 6 where chamber pressure recorded during nitrogen diffusion measurements is shown in a semi-logarithmic scale. Such a plot should be linear except for the initial and final stages of the diffusion process, and the intermediate part of the plot can be used for curve fitting.

Two curves were calculated with the effective diffusion coefficient of  $6.2 \times 10^{-11}$  m<sup>2</sup>/s and  $6.8 \times 10^{-11}$  m<sup>2</sup>/s. For each of them, during a period from 3 to 20 hours (linear part of the curve) an average difference between calculated and measured pressures was established in the same manner as one calculates standard deviation, i.e., a square root of the sum of the squares of

the differences between calculated and measured pressures divided by  $n$ , where  $n$  is the number of measurements. The average differences were 499 and 439 Pa for each of calculated curves respectively. In relation to the average pressure of 122700 Pa, these differences were 0.41 and 0.36%.

For all practical purposes, each of the curves whether calculated with effective diffusion coefficient of  $6.2 \times 10^{-11} \text{ m}^2/\text{s}$  or  $6.8 \times 10^{-11} \text{ m}^2/\text{s}$  is equally probable. Thus, despite high precision of pressure, volume and thickness measurements, as shown above, determination of the effective diffusion may not be reliable to more than one significant figure.

### COMMENTS ON LIMITATION OF THIS METHOD

The limitations of this test method stem from the limitations in the validity of the governing equations, which assume:

- (a) Constant effective diffusion coefficient (since diffusion coefficient is a ratio of permeability to mass storage factor this assumption implies that both permeability and mass storage factor remain constant)
- (b) For a given change in partial pressure of the gas the change in mass (mass storage factor) follows ideal gas equation.
- (c) Predominantly one directional transfer in presence of unidirectional gradient of pressure, i.e., predominantly isotropic structure
- (d) Specimen volume is large enough to consider material property as independent of the volume tested.

Concern (a) requires a fundamental analysis of gas sorption and penetration into polymer matrix, and is not easy to address. To address concern (b), two computer programs were written. One program used separate values of gas permeability and mass storage factors, and allowed modification of each of the values. The other program used an effective diffusion coefficient, i.e., a ratio of these two variables and ideal gas law to calculate the mass storage factor. For the measurements examined in this work, namely nitrogen and oxygen diffusion in polystyrene, both programs gave the same results.

Concerns (c) and (d) were partly addressed by Shankland [3] who postulated that the specimen thickness must exceed 20 cell-layers, i.e., the thickness of cellular plastic specimens must not be less than 3–5 mm.

### CONCLUDING REMARKS

This paper presented an iterative procedure to calculate gas diffusion coefficients involving two sets of measurements:

1. The rate of gas diffusion in cellular plastics
2. Changes in thermal resistivity with time

Both heat and gas flow measurements were performed on thin material layers, not only to reduce the measurement period but also to prepare test specimens with structure as uniform as possible.

The procedure was applied to a core of an extruded polystyrene board and three effective diffusion coefficients were determined. These coefficients were then used for calculations and compared with other independent experiments, see Figures 5-8. One may observe that the values of oxygen, nitrogen and CFC diffusion coefficients determined with the proposed procedure were representative for the examined material and can be used in evaluation of long-term thermal resistance.

### ACKNOWLEDGEMENTS

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