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Interlaboratory Comparison of the ASTM Standard Test Methods for Water Vapor Transmission of Materials (E 96-95)

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ABSTRACT: An interlaboratory comparison was initiated by the ASTM C16 Committee on Thermal Insulation to determine the precision of the test method prescribed by ASTM Standard Test Methods for Water Vapor Transmission of Materials (E 96-95). Expanded polystyrene board was chosen as the test material. Nine laboratories completed the measurements according to the dry cup and the wet cup procedures prescribed by the standard. Statistical analyses of the data resulted in the following precision statement for the water vapor permeability:

Dry cup method:

Permeability of material = $3.1\text{E-}12 \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$

Repeatability standard deviation (within a laboratory) = $1.6\text{E-}13 \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$

Reproducibility standard deviation (between laboratories) = $4.7\text{E-}13 \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$

Wet cup method:

Permeability of material = $3.4\text{E-}12 \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$

Repeatability standard deviation (within a laboratory) = $1.7\text{E-}13 \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$

Reproducibility standard deviation (between laboratories) = $4.1\text{E-}13 \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$

A specific procedure was uniformly used to analyze the data and to calculate the permeability of the test material from the raw data provided by the participating laboratories. Corrections were made for the resistances offered by the still air layer inside the cup and by the specimen surfaces and for the edge masking details.

KEYWORDS: water vapor transmission, permeability, permance, relative humidity, desiccant method, water method, repeatability, reproducibility

Water vapor permance is probably the most commonly used hygrothermal property that characterizes building materials. Almost all insulation material specifications in North America refer to this property as a performance parameter [1]. The property originates from the water vapor transport equation:

$$\dot{m}_v = -\delta_p \text{grad } p_v \quad (1)$$

where

\dot{m}_v = water vapor transmission rate, $\text{kg m}^{-2} \text{ s}^{-1}$

δ_p = water vapor permeability of the medium, $\text{kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$

p_v = water vapor pressure, Pa

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The above equation is derived from Fick's law, which states that the density of diffusion flow rate of any substance at a given point in a medium is directly proportional to its concentration gradient at that point, at constant temperature. In building physics applications, the water vapor pressure being significantly lower than the atmospheric pressure, water vapor is assumed to follow the ideal gas equation and hence the concentration is substituted by the vapor pressure in Eq 1.

Traditionally, building physicists have used Eq 1 directly to measure the water vapor permeabilities of building materials. There has been very little change in the principle of the experimental procedure during the past six decades. In 1963, Joy and Wilson [2] thus wrote:

The simplest and most widely used test of the rate of water vapor transmission is the dish or the cup method. The specimen is sealed to the mouth of an impermeable test dish containing water or a desiccant, and placed in a controlled atmosphere. Periodic weighing of the assembly determines when a steady rate of mass loss or gain is attained, and this final rate, continued long enough to ensure its validity, is the test result. . . .

ASTM had standardized two dish methods, as early as 1954: E 96 for thin sheets and C 355 (discontinued, replaced by E 96) for materials, such as those used in building construction, tested in a thickness greater than 3 mm. Currently, the ASTM Standard Test Methods for Water Vapor Transmission of Materials E 96-95 is followed in Canada and the U.S. This standard prescribes two test procedures: a desiccant method and a water method known respectively as the dry cup and the wet cup methods. In the former, the cup contains a desiccant (usually anhydrous calcium chloride to create an atmosphere of 0% relative humidity) and in the latter, distilled water to maintain 100% relative humidity. In either procedure the cup is placed in a chamber maintained at 50% relative humidity at a constant temperature to determine the steady-state water vapor transmission across the test specimen attached to the mouth of the cup.

The procedures appear to be simple in principle. But as Tye [3] commented,

Method E 96, for example, contains estimates of probable levels of uncertainty due to a number of factors. These estimates differ depending on the individual procedure and on the type of the material retarder and its thickness. In general, the maximum errors (worst case) due to a combination of known factors are estimated to be the order of ± 25 to 30% while $\pm 10\%$ is considered

to be an acceptable norm. However, in some of the earlier measurements the results indicated that uncertainties of the order of ten times and greater than the worst case were not uncommon.

The method of sealing the specimen to the mouth of the cup has a major influence on the results from the measurements. Also, as explained below, differences in the procedure used by individual laboratories to analyze the data result in differences in the reported values for permeances and permeabilities.

The standard prescribes a procedure for the data analysis. A least-squares regression analysis of the change in mass as a function of time is used to determine the rate of water vapor transmission at the steady state. However, in the current standard, the rate so calculated is assumed to correspond only to the resistance offered by the test specimen. In fact, the experimental setup imposes four vapor resistances [4,5] in series between the content of the cup (the desiccant or distilled water) and the chamber:

1. resistance offered by layer of still air in cup,
2. resistance offered by surface of specimen inside cup,
3. resistance offered by test specimen, and
4. resistance offered by surface of specimen outside cup.

While some of the laboratories make corrections [5] for these resistances, others do not. This results in large differences in the reported values for permeances and permeabilities, especially if the test material is highly permeable.

Yet another type of correction that cannot always be neglected is the buoyancy correction. The duration of one set of measurements can be many days or weeks. The atmospheric pressure may change significantly during such periods. If the test specimen is highly vapor resistant, the changes in mass due to the vapor transport may be overshadowed by the apparent gravimetric changes observed.

Another reason for differences in reported values is whether or not the results are corrected for the edge masking details. Wax is recommended for sealing the test specimen to the mouth of the cup. Depending on the size and shape of the cup and the technique of the sealing, various fractions of the surface areas of the test specimens are masked at the edge. This results not only in a reduction in the effective test area but also introduces an alternate path for vapor diffusion within the specimen. Hence, the measured water vapor transmission rates should be corrected [2]. In practice, differences between 5% and 20% may result from this correction.

There is a general consensus that ASTM E 96 and similar methods used elsewhere need a thorough revision with attention paid specifically to specimen assembly, choice of temperature and humidity conditions and the subsequent evaluation and interpretation of the results [3]. A task group within the ASTM C16 Committee on Thermal Insulation is currently reviewing a draft proposal that addresses some of these issues; an interlaboratory comparison was initiated in 1995 to investigate the influence of various parameters. The project has now been completed and this paper reports the results.

Work Plan for the Interlaboratory Comparison

A set of 60 × 60 cm samples of expanded polystyrene boards (≈25 mm thick) was prepared at the Institute for Research in Construction from a carefully chosen batch of the product. The average density of the material was ≈15.8 kg m⁻³. One or two of these

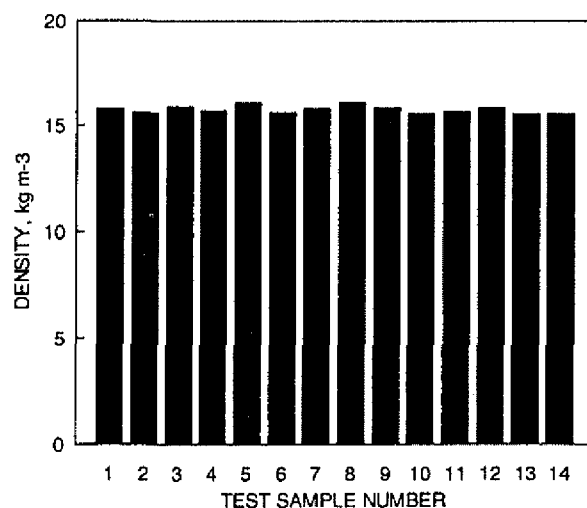


FIG. 1—Densities of 14 test samples of expanded polystyrene insulation used for the interlaboratory comparison.

samples were sent to 11 participating laboratories. The distribution of densities of the test samples is shown in Fig. 1.

The work plan shown in Fig. 2 was sent to each participating laboratory. Ten of the participating laboratories completed and reported their measurements according to the work plan. One laboratory withdrew from the project.

Results

The freedom allowed by ASTM E 96 resulted in a variety of experimental parameters to be chosen by the participating laboratories. The shape of test specimens, for example, was circular for eight laboratories, square for one and oval for another. The test area varied between 45 and 650 cm². The thickness of the still air layer ranged between 6 and 19 mm. The sealing technique and edge masking resulted in corrections between 0 and 21%.

All the raw data were forwarded to the Institute for Research in Construction and used for analysis using a uniform procedure. One of the primary criteria was that each set of data (time versus mass change) on any test specimen yield a linear correlation coefficient between 0.998 and 1 to confirm the attainment of steady state. Results from one laboratory failed to attain this and were not included in subsequent analysis. All corrections, such as those for the resistances offered by the still air layer in the cup and the surfaces of specimen and that for errors due to edge masking, were made according to the information provided by each laboratory using procedures recommended in the literature [2,4,5]. The measured mass changes were large enough that buoyancy corrections were insignificant and hence unnecessary. The results are listed in Table 1.

Averaging of all the data in Table 1 gives the following values:

Water vapor permeability from dry cup measurements

$$= (3.1 \pm 0.5) \text{E-12 kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$$

Water vapor permeability from wet cup measurements

$$= (3.7 \pm 1.3) \text{E-12 kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$$

The results for the wet cup measurements from Laboratory 4

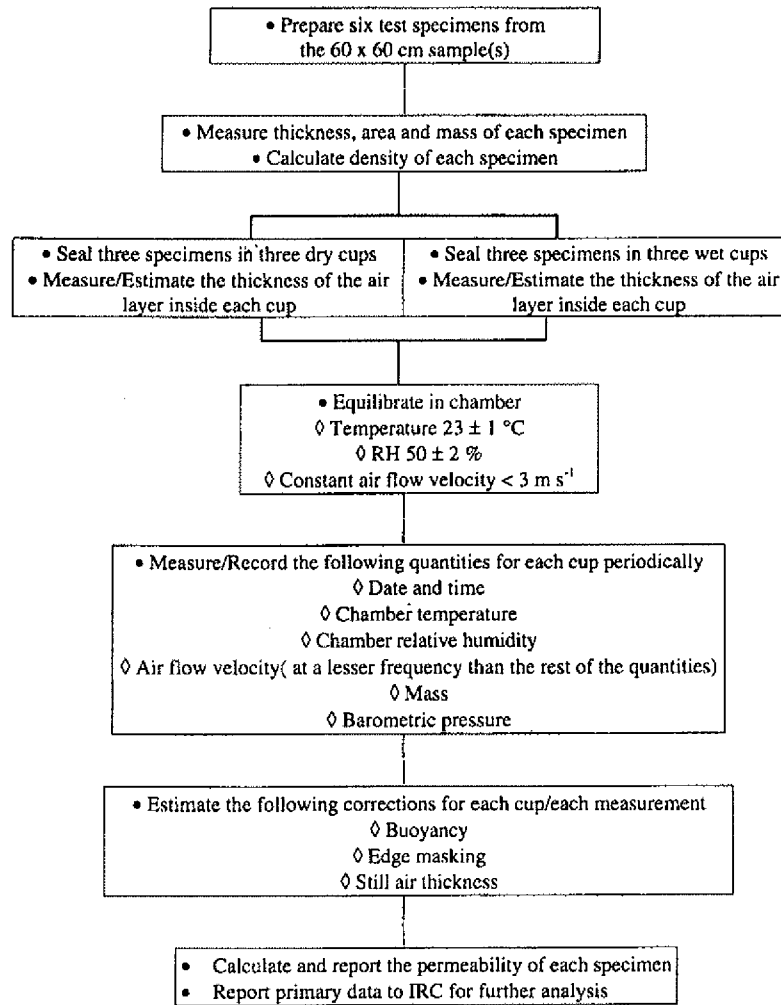


FIG. 2—Work plan.

TABLE 1—The permeability, $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$, of the expanded polystyrene material from nine laboratories.

Laboratory	Dry Cup Results	Wet Cup Results	Laboratory	Dry Cup Results	Wet Cup Results
1	2.54E-12	2.90E-12	6	3.26E-12	2.61E-12
	2.46E-12	3.14E-12		3.38E-12	2.82E-12
	2.21E-12	2.94E-12		3.29E-12	2.80E-12
2	2.65E-12	3.50E-12	7	3.05E-12	3.53E-12
	2.87E-12	3.46E-12		3.72E-12	3.18E-12
	2.68E-12	3.52E-12		3.33E-12	3.41E-12
3	3.79E-12	4.23E-12	8	3.76E-12	3.30E-12
	3.49E-12	3.76E-12		3.53E-12	3.42E-12
	3.65E-12	3.65E-12		3.87E-12	3.29E-12
4	2.77E-12	5.69E-12	9	3.24E-12	3.75E-12
	2.73E-12	5.85E-12		3.48E-12	3.97E-12
	2.69E-12	8.92E-12		3.26E-12	3.67E-12
5	2.67E-12	3.32E-12	
	2.66E-12	3.29E-12	
	2.79E-12	2.97E-12	

are solely responsible for the large standard deviation for this category. If these results, based on their relatively large deviations, are considered as outliers, then:

$$\text{Water vapor permeability from wet cup measurements} \\ = (3.4 \pm 0.4)\text{E-12 kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$$

The standard deviations from both sets of measurements, 15% for the dry cup measurements and 11% for the wet cup measurements, fall well within those obtained from interlaboratory comparisons. For example, in the recently concluded "Intercomparison on Measurements of Water Vapour Permeability," in which 13 European laboratories participated, measurements on extruded polystyrene and particle board specimens yielded 19% and 21% deviations from the average value [6].

Significance of the Corrections

It is important that all applicable corrections be made to all measurements. The procedures for making these corrections are found in the literature and are summarized below.

Buoyancy Correction—The following equation can be used for buoyancy corrections [7]:

$$\frac{m_2}{m_1} = 1 + \frac{\rho_a(\rho_1 - \rho_2)}{\rho_1(\rho_2 - \rho_a)} \quad (2)$$

where

$$\begin{aligned} m_1 &= \text{mass recorded by balance, kg} \\ m_2 &= \text{mass corrected for buoyancy effect, kg} \\ \rho_a &= \text{density of air, kg m}^{-3} \end{aligned}$$

ρ_1 = density of material of balance weights, kg m^{-3}
 ρ_2 = bulk density of test assembly, kg m^{-3}

The density of air can be calculated using the ideal gas law for the measured atmospheric pressure and ambient temperature.

Corrections for Resistances Due to Still Air Layer and Specimen Surface—If the thickness of the still air layer is known, the corresponding vapor permeance and hence the resistance can be calculated using the following equation for permeability [8].

$$\delta_a = \frac{2.306 \times 10^{-5} P_o}{R_v T P} \left(\frac{T}{273.15} \right)^{1.81} \quad (3)$$

where

δ_a = permeability of still air, $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$
 T = temperature, K
 P = ambient pressure, Pa
 P_o = standard atmospheric pressure, i.e., 101 325 Pa
 R_v = the ideal gas constant for water, i.e., $461.5 \text{ J K}^{-1} \text{kg}^{-1}$

In the absence of any measured data, the surface resistances may be approximated using Lewis's relation [9]. According to Hansen and Lund [4], for cup methods that follow the ASTM Standard, the total surface resistance should be $\approx 4 \times 10^7 \text{ Pa s m}^2 \text{kg}^{-1}$.

Edge Mask Correction—Joy and Wilson [2] have recommended the following equation for the excess water vapor transmission (WVT) due to edge masking:

$$\text{Percent excess WVT} = \frac{400t}{\pi S} \log_e \left(\frac{2}{1 + e^{-(2\pi b/t)}} \right) \quad (4)$$

where

t = specimen thickness, m
 b = width of masked edge, m
 S = four times test area divided by perimeter, m

As shown by Lackey et al. [5], the buoyancy correction is important where the measured mass changes are in the range of 0 to 100 mg. For the present specimens the changes were in the range of 2 to 30 g and thus the buoyancy correction was always insignificant. However, other corrections were often significant as shown in the following example.

The results from the wet cup measurements from Laboratories 7 and 8 are used for the calculations and included in Table 2. These results represent the two extremes in terms of the edge masking correction. The results from Laboratory 7 include an excess WVT

TABLE 2—Significance of various corrections for calculating the permeability, $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$.

Laboratory No.	Specimen No.	All Corrections Made	No Correction Made	Corrections Made Only for Resistances
7	1	3.53E-12	4.39E-12	4.50E-12
	2	3.18E-12	3.97E-12	4.06E-12
	3	3.41E-12	4.25E-12	4.35E-12
	Average	3.37E-12	4.20E-12	4.30E-12
8	1	3.30E-12	3.26E-12	3.30E-12
	2	3.42E-12	3.37E-12	3.42E-12
	3	3.29E-12	3.24E-12	3.29E-12
	Average	3.34E-12	3.29E-12	3.34E-12

of 21% while those from Laboratory 8 have no edge masking correction. The thickness of the still air layer for Laboratory 7 is 21 mm while that for Laboratory 8 is 15 mm.

The differences between the numbers in columns 3 and 4 for Laboratory 7 are very large ($\approx 25\%$) in comparison with the standard deviation of 11% stated above. Furthermore, the average in column 3, where all applicable corrections are made, is nearly equal to the grand average from all the laboratories and the average from Laboratory 8. This confirms the significance of the corrections.

For the present test material, the differences between the numbers in columns 4 and 5 are not large. This is not always the case. For example, if a material is highly permeable, the corrections for the additional resistances become significant. This is illustrated below using the results on a specimen of a medium density glass fiber insulation.

Test Results on a Medium Density Glass Fiber Insulation Specimen

Water vapor transmission rate	$= 2.27 \times 10^{-6} \text{ kg m}^{-2} \text{s}^{-1}$
Permeance (uncorrected)	$= 3.85 \times 10^{-9} \text{ kg m}^{-2} \text{s}^{-1} \text{Pa}^{-1}$
Thickness of still air	$= 15 \text{ mm}$
Permeance (corrected for still air layer)	$= 5.46 \times 10^{-9} \text{ kg m}^{-2} \text{s}^{-1} \text{Pa}^{-1}$
Total surface resistances	$= 4 \times 10^7 \text{ Pa s m}^2 \text{kg}^{-1}$
permeance (corrected for still air layer and surface resistances)	$= 6.98 \times 10^{-9} \text{ kg m}^{-2} \text{s}^{-1} \text{Pa}^{-1}$

If the corrections are not made, the error in the calculated permeance for this test specimen would have been $\approx 45\%$.

Statistical Analysis of the Data

The results in Table 1 were analyzed according to the ASTM Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method (E 691-92). The results are listed in Tables 3, 4, and 5. The symbols have the following meanings:

- \bar{x} = Cell average or the average from one laboratory, $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$
- s = Cell standard deviation, or the standard deviation for one laboratory, $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$
- $\bar{\bar{x}}$ = Average of the cell averages, $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$
- $s_{\bar{x}}$ = Standard deviation of the cell averages, $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$
- d = Cell deviation or the difference ($\bar{x} - \bar{\bar{x}}$), $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$
- s_r = Repeatability standard deviation (within a laboratory), $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$
- s_R = Reproducibility standard deviation (between laboratories), $\text{kg m}^{-1} \text{s}^{-1} \text{Pa}^{-1}$
- h = Between-laboratory consistency statistic, [—]
- k = Within-laboratory consistency statistic, [—]

The quantities h and k , the between-laboratory and the within-laboratory consistency statistics, in Tables 3 and 4 suggest the following: The results from Laboratory 7 in Table 3 should be treated as marginal, the k value is very close to the critical value of 2.09, and the results are just acceptable.

The estimated values for h and k for Laboratory 4 in Table 4

Center for Applied Engineering, Inc. (St. Petersburg), The Dow Chemical Company (Granville), Holometrix (Bedford), Mountain Technical Center (Littleton), The Institute for Research in Construction, The National Research Council of Canada (Ottawa),² Pittsburgh Corning Corporation (Pittsburgh) and Styrochem International, Inc. (Fort Worth). The contribution from Mr. John Lackey of the NRCC for the organization of the project deserves special recognition.

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² Two separate series of measurements were done at the NRCC.