PROCEDURES FOR THE EVALUATION OF SHEET MEMBRANE WATERPROOFING

Charles J. Korhonen, James S. Buska, Edel R. Cortez and Alan R. Greatorex

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Sheet membrane waterproofing had been used to protect bridge decks against water and deicing salts by transportation agencies in New England for more than two decades. Though such membranes have proven useful at extending the useful life of bridge decks, there are no convenient methods to evaluate one membrane against another. This report details the genesis of blisters, a major problem for membranes, and defines test procedures to evaluate sheet membranes based on their ability to adhere to concrete, accommodate strain, resist puncturing, and pass water vapor. The results of these tests allow an engineer to compare sheet membranes based on material properties but they, alone, cannot be used to predict how well a membrane will perform in practice. Because a laboratory environment does not reflect the complex combination of forces and deterioration mechanisms a membrane is exposed to in the field, a follow-on study of the installation/design process and long-term performance of membranes in actual bridges needs to be conducted. This report provides a needed step toward the ability to predict sheet membrane service life.

**Key Words:** bridge decks, sheet membrane, waterproofing, deicing salts, blisters, adhesion, tensile strength, puncture resistance, water vapor permeance

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PREFACE

This report was prepared by Charles J. Korhonen, James S. Buska, and Edel R. Cortez, Research Civil Engineers, and Alan R. Greatorex, Civil Engineering Technician, of the Civil Engineering Research Division, U.S. Army Cold Regions Research and Engineering Laboratory.

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Procedures for the Evaluation of Sheet Membrane Waterproofing

CHARLES J. KORHONEN, JAMES S. BUSKA, EDEL R. CORTEZ, AND ALAN R. GREATOREX

INTRODUCTION

At the request of the New England Transportation Consortium (NETC), the U.S. Army Cold Regions Research and Engineering Laboratory (CRREL) conducted laboratory studies from March 1998 to March 1999 to standardize procedures to evaluate bridge deck membranes. This report presents the results of these studies and completes the requirements of NETC Project Number 94-3.

Background

Waterproofing membranes have been used to protect concrete bridge decks by transportation agencies in New England for more than two decades. Over the years, membranes have proved useful for preventing water and deicing salts from penetrating the concrete and corroding the embedded reinforcing steel. Frascoia (1983), in his 11-yr field exposure study of 33 membrane systems, demonstrated that brushed-on coatings of coal tar emulsion significantly reduced the ingress of chloride into concrete, though not as efficiently as sheet systems. Unprotected bridge decks absorbed 6.97 lb/yd² (4.11 kg/m²) of chloride ions in the top inch of concrete, but the decks absorbed only 0.65 lb/yd³ (0.38 kg/m³) when the concrete was coated with tar emulsion and 0.50 lb/yd³ (0.30 kg/m³) when a sheet membrane was used on top of the concrete. However, tar emulsions have not provided consistent protection and were judged by the Vermont Agency of Transportation as unacceptable. Because chloride does not seriously corrode rebar until it reaches at least 1.30 lb/yd³ (0.77 kg/m³) (Lewis 1962, Clear 1974), an interlayer waterproofer should thus be an improvement and considered as an important bridge element.

Bukovatz et al. (1983) provided a similar endorsement of waterproofing systems when they characterized the performance of sheet and liquid membranes as satisfactory after 12 to 16 years of field exposure. Wojakowski and Hossam (1995) later reevaluated six of the eight membranes studied by Bukovatz et al., concluding that the general performance of the membranes had decreased significantly. After 25 years of service, they estimated that the lives of some systems had been exhausted. Frascoia (1993) projected that the membranes he studied would provide protection from salt contamination for more than 50 years.

A membrane will protect a deck only if it is installed properly, stays intact, and remains firmly bonded to the deck; cracked or poorly bonded membranes can lead to serious roadway deterioration such as cracking and potholing. Construction is a crucial time in the life of a membrane, because it is during construction that most problems begin. For example, membranes are subject to abrasion damage from foot and vehicle traffic, puncture from dropped objects and rocks pressed into the membrane, and poor adhesion due to inadequate workmanship, inclement weather or material defects. Poor adhesion can also result from the deck surface being too rough or uneven. Whatever the cause, inadequately installed membranes tend to puncture, blister, and crack at some point during their service life, which weakens a membrane to chloride and moisture penetration and ultimately results in failure of the overlay pavement. In turn, this accelerates deck deterioration and presents rough surfaces to the motoring public.
Objectives

Though field tests have proven that membranes reduce chloride contamination of underlying concrete, there are problem areas where improvements in test procedures or materials are needed. If a membrane cannot be fully adhered to the deck, or it somehow becomes damaged during construction or is unable to resist splitting when cracks develop in the underlying deck or bituminous overlay, moisture and chlorides can leak through the system and accelerate bridge deterioration. The objectives of this work were to develop laboratory tests for evaluating sheet membrane waterproofing for their ability to resist cracking, blistering, and puncturing. ASTM lists a number of tests to evaluate various engineering properties of tape, rubber, roofing, plastics, and geomembranes. The problem is that there is no group of standards, or ways to interpret them, that all manufacturers follow when reporting performance data for their products. As a result it is difficult, if not impossible, to rate one membrane against another based on manufacturer-supplied data. Our plan was to review these and other literature to develop a set of testing standards specific to the above objectives.

Approach

NETC developed a list of sheet membranes that have been used on bridge decks in New England. From that list we invited suppliers of membranes to participate in this study by providing materials and by making test samples. (Several suppliers of liquid membranes were also interested in participating but were not accommodated, because testing liquid membranes was not within the scope of this study.) The intent of this work was to recommend tests to evaluate one membrane against another. We acknowledged that until a systematic field test is conducted, these laboratory tests could not reliably predict expected service life, as laboratory tests do not simulate field conditions and, therefore, only suggest possible outcomes in the field.

This project subjected sheet membranes from the six manufacturers shown in Table 1 to the following four tests:

- **Adhesion**: to evaluate the adhesion developed between a membrane and a concrete substrate.
- **Tensile strength and elongation**: to determine how well a membrane can resist and accommodate movement of the concrete deck.
- **Puncture resistance**: to measure the resistance of a membrane to rock puncture.
- **Water vapor permeance**: to determine how easily water vapor can pass through a membrane.

For reference and general interest, Appendix A presents technical data from manufacturers' brochures for each membrane evaluated in this project.

**ADHESION**

Lack of adhesion is considered to be the leading cause of membrane blistering. This study reviewed current testing standards, such as ASTM C794, D903, and D1000, and developed one that could be used as the standard by which to evaluate the ability of sheet membranes to adhere to concrete.

**Procedure**

Adhesion was measured by peeling strips of membrane off mortar. The test consisted of adhering membranes to carefully prepared mortar surfaces, cutting the membrane into strips, and applying a tensile load at a constant rate of extension until each strip peeled off the mortar a predetermined distance. Test specimens were prepared, as shown in Figure 1, according to manufacturers' recommendations. Two sets of test specimens for each of the six membrane types were constructed: CRREL made three specimens and the membrane supplier made three. Making two sets of samples helped to determine if choice of applicator influenced results. The specimens were prepared as follows:

- At room temperature, mix one weight of Type I portland cement with two weights of Ottawa sand (20–30 grade) according to ASTM C305.
- Cast mortar into 6- × 6- × 21-in. (15- × 15- × 53-cm) molds and cover with sheet of plastic.
- After 24 hours, strip molds and cure mortar beams in room-temperature limewater for 14 days.
- Cut beams into 6- x 6.38-in. (15.2- x 16.2-cm) slices.
- Sand slices (slabs) with 24-grit silicon-carbide sandpaper until surface is flat and all saw marks are removed.
- Oven dry the mortar slabs at 220°F (104°C) for 24 hours.
- Clean sanded surface with dry, stiff fiber bristle brush.
- Place 0.75-in. wide strip of tape across one end of slab.
- Apply primer to the test surface.
- Allow primer to cure to a tack-free finish.
- Apply membrane according to manufacturers’ instructions.
- Condition specimens at approximately 70°F (21°C) and 50% RH (relative humidity) for a minimum of 14 days.
- Cut membrane into five 1-in.- (2.5-cm) wide strips through to the mortar with a sharp razor knife.
- Start cuts 0.50-in. (1.3 cm) from edge of slab.

Figure 2 shows the test setup. Five strips of membrane were peeled off each slab back at an angle of 180° at a grip separation rate of 4 in. (10.2 cm)/min. Force and grip displacement were recorded for each strip. Slippage in the grips and membrane stretching are discounted, grip displacement is exactly twice membrane displacement. Before discussing the significance of the adhesion test data, we will first consider the mechanics of blistering.

**Blister mechanics**

In a related study, Korhonen (1986) pointed out that roof membrane blisters develop from voids built into a roof during construction. There is no reason to suspect that bridge blisters are any different. They probably are caused by the expansion of air pockets inadvertently trapped between the membrane and the concrete deck during construction. Roughness of the concrete deck, unevenly applied or inadequately cured primer, debris, and moisture are among a number of reasons that can impair the adhesion of a membrane to a deck and lead to blister-causing voids. On the other hand, a perfectly adhered membrane (if it exists) cannot blister.

Fortunately, a membrane does not have to be perfectly adhered to a deck. Mathematically, it can be shown that some voids are acceptable. When blisters form, they appear as slightly bloated humps—in the membrane or the overlying pavement—several inches to a foot or two in diameter. They often occur soon after the membrane is laid or immediately after hot-mix pavement is placed on top of the membrane. As eq 1 shows, growth happens only when the air inside a void is heated sufficiently to push the overburden upward and peel it off the deck:

\[ F = (PA - WA)/L \]  

where \( F \) = membrane-to-deck peel strength  
\( P \) = internal pressure
a. Installing a membrane.

b. Peeling membrane strip from mortar slab.

Figure 2. Typical installation and adhesion test setup.
\[ A = \text{area of void} \quad (\pi r^2) \]
\[ W = \text{overburden (weight of material on top of blister)} \]
\[ L = \text{perimeter of void}. \]

Equation 2 explains that the smaller the void, the less likely it is to develop into a blister:

\[ r = \frac{2F}{(P - W)} \quad (2) \]

where \( r \) is void radius. That is, it requires more internal pressure (heat) to expand a small void than to expand a large one.

Figure 3, developed from eq 2, illustrates this concept. It consists of four graphs, each composed of three curves, where each curve represents peel strength plotted against temperature and critical size. Each graph defines the smallest void expected to blister. For example, if an air pocket beneath a membrane adhered to a deck at 5 lbf/in. (875 N/m) is heated from 70°F to 140°F (24°C to 67°C), a 5.2-in. (13.2-cm) radius would be the smallest void that could blister (Fig. 3a). However, if the air beneath the membrane is continually water saturated, the critical void would reduce to 2.25 in. (5.7-cm) radius (Fig. 3b). Of course, higher bond strengths are more resistant to blistering, but one must realize that heat, the driving force of blisters, softens the adhesive and diminishes peel strength. Thus, the 5-lbf/in. force used in the above analogy is considered conservative, even though some membranes adhere more tightly to concrete at room temperature.

The situation changes as soon as the membrane is topped with hot pavement. In this case the void immediately heats up to 250°F (146°C) or more and
its overburden increases more than 20-fold (a membrane weighs between 0.002 to 0.008 lb/in.² whereas 2-in.-thick asphalt pavement weighs approximately 0.168 lb/in.²). In this situation we see that the critical size changes from a 2.1-in. (5.3-cm) radius when the void space is dry (Fig. 3c) to a 0.30-in. radius when it is wet (Fig. 3d). Moreover, blisters do not just expand once, they continually increase in size. Korhonen (1986) found this to be true for roof blisters as did Hironaka and Holland (1986) for pavement blisters. Thus, once a blister initiates, no matter how small it may be, it eventually grows large enough to become a big problem.

Though Figure 3 represents idealized situations (a blister is not rigid and self-contained), clearly a nonporous membrane exposed to the sun will remain blisterless if its voids are smaller than 5.5 in. (14 cm) across (Fig. 3b). When exposed to the intense heat of freshly laid pavement, approximately quarter-sized voids (0.9 in., or 2.4 cm) (Fig. 3d) can lead to problems. Other scenarios are possible for blisters but the quarter coin size should be useful as a rule of thumb for bridge inspectors to distinguish when a membrane is being inadequately adhered to a deck. A permeable membrane can reduce blistering by allowing pressure build up to escape through the membrane. However, the section on water vapor permeance reveals that the membranes in this study were not very breathable.

Results and discussion

The entire data set for the adhesion tests consists of force-displacement diagrams for 157 strips of membrane peeled off mortar slabs (App. B). We will not discuss each diagram but, rather, summarize them in Figure 4 and make specific references to them in the following text to give the reader a sense of their significance. The reader is encouraged to peruse Appendix B for added detail.

Figure 4 shows six force-displacement diagrams, one for each membrane type where each is composed of two curves (except for Figure 4f, where supplier samples were not available). As can be seen by the difference between the two curves in each graph, the choice of applicator can influence results. Each curve represents the average of up to 15 strips peeled from three samples fabricated by the membrane supplier compared to three done by CRREL. The Figure 4 curves reveal that testing was done in two stages. In the first stage, approximately 2 to 2.25 in. (5.7 cm) of the membrane was peeled off the slabs. The membrane was then unloaded, repositioned in the grips—as the grips reached the end of their movement—and peeled approximately another 2 in. (5 cm). In interpreting the curves, one should recognize that the pulling force for each stage gradually built up, as the membrane and adhesive stretched and as the membrane seated in the grips, until the force became large enough to progressively peel the membrane from the slab.

The shape of each curve shows that adhesion is a complex issue, difficult to describe with just one number. Should a maximum, minimum, or average force be used to describe adhesion? Maximum values can be considered as the very best adhesion that one can hope to expect. It could be argued that the resistance offered by a membrane just prior to the onset of progressive peeling sometimes mimics that of a membrane against the initiation of a blister on a bridge. For some membranes in this study, however, progressive peeling did not occur until the pulling force peaked; thereafter, peeling occurred at a lower force (Fig. 4a, 4b, 4c and individual results for Polyguard and Soprema, App. B). This is not unlike what occurs on bridges where, once growth is initiated, blisters seem to expand quite rapidly for a while. For other membranes, peak forces did not develop at all (Fig. 4d, 4e and 4f) near the end of the test (Fig. 4b and 4c). Average values, on the other hand, show a typical adhesion that can be expected. Averages dampen out any of the extreme values during testing and usually provide a reasonable basis of comparison. However, by considering the discussion on blister mechanics, clearly neither maximum nor average values are adequate, because the root cause of all blisters is poor adhesion. Therefore, minimum values are a revealing test result, because a blister simply cannot form unless a membrane is poorly bonded, at least in spots.

With the foregoing in mind, Table 2 was developed from Appendix B to compare the maximum, average, and minimum adhesion values measured in this study. (The values developed from the CRREL-made samples are differentiated from those made by the supplier.) The maximum values in Table 2 were based on the entire loading curve of each strip, whereas the average and minimum values came from the center portion of each testing stage (i.e., from approximately 1 to 2 in. of stage 1 and 3 to 4 in. of stage 2). Using only the center portion avoided effects caused by the start up or end of each test. As can be seen, the Polyguard and Soprema membranes have the best adhesion values when either maximum or average values are considered. However, the situation changes when minimum values are considered.
Figure 4. Adhesion test results. Each curve is the average of up to 15 test strips. Surprisingly, the CRREL samples consistently developed high overall, but low individual, adhesion values (Fig. 4 vs. App. B).

Here, the Protecto Wrap and W.R. Grace membranes become the membranes with the least potential problems. They exhibit moderate, but very uniform, adhesion values with little indication of weak spots (Fig. 4a and 4d). Interestingly, the Protecto Wrap membrane seemed the least influenced by the source of the samples for testing. This uniformity suggests that this membrane would provide a consistent adhesion in the field from job to job and contractor to contractor.

TENSILE STRENGTH AND ELONGATION

Waterproofing membranes must be able to span active cracks in a deck, especially at low temperatures, when cracks widen most. To do this, a mem-
Table 2. Comparison of adhesion values.

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Average</th>
<th>Maximum</th>
<th>Minimum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyguard</td>
<td>11.20</td>
<td>19.42</td>
<td>6.75</td>
</tr>
<tr>
<td></td>
<td>14.82</td>
<td>43.05</td>
<td>0.68</td>
</tr>
<tr>
<td>Soprema</td>
<td>9.54</td>
<td>29.02</td>
<td>1.34</td>
</tr>
<tr>
<td></td>
<td>11.13</td>
<td>36.32</td>
<td>0.00</td>
</tr>
<tr>
<td>NEI</td>
<td>†</td>
<td>†</td>
<td>†</td>
</tr>
<tr>
<td></td>
<td>10.19</td>
<td>14.98</td>
<td>5.23</td>
</tr>
<tr>
<td>W.R. Grace</td>
<td>6.39</td>
<td>11.46</td>
<td>2.87</td>
</tr>
<tr>
<td></td>
<td>10.52</td>
<td>17.93</td>
<td>4.18</td>
</tr>
<tr>
<td>Protecto Wrap</td>
<td>8.35</td>
<td>14.70</td>
<td>5.23</td>
</tr>
<tr>
<td></td>
<td>7.05</td>
<td>9.25</td>
<td>4.45</td>
</tr>
<tr>
<td>Royston</td>
<td>3.17</td>
<td>6.97</td>
<td>1.71</td>
</tr>
<tr>
<td></td>
<td>4.93</td>
<td>9.61</td>
<td>2.88</td>
</tr>
</tbody>
</table>

*From Appendix B.
†Samples not available.
The top row of readings for each material is from the supplier-made samples and the bottom row from the CRREL samples.

brane must either be strong enough to resist the tensile force caused by crack movement or stretchable enough to accommodate it. This study reviewed ASTM standards D412, D638, D882, D2523, and D4885 before developing a testing protocol to evaluate the ability of waterproofing membranes to resist splitting and elongation at various temperatures, including below freezing.

Procedure
Tensile strength and elongation were determined at five temperatures (70°F, 40°F, 23°F, 14°F and -4°F [21°C, 4°C, -5°C, -10°C, -20°C]), by pulling apart three dogbone-shaped specimens per membrane (Fig. 5) at a grip separation rate of 4 in./min. Prior to testing, the specimens were conditioned one hour at the appropriate test temperature. Figure 6 shows the test setup.

Results and discussion
Test results for tensile strength and elongation are shown in Appendix C and Table 3. Appendix C shows load–strain curves for each membrane tested at five temperatures. We will not discuss each curve, but the reader is encouraged to review Appendix C to understand how the measurements were obtained and to get a sense for how each membrane behaved. Table 3 was developed from these data to directly compare ultimate strength and elongation of the membranes as a function of temperature.

Table 3 shows that, though the membranes are not all alike in strength, they all tend to become stronger at lower temperatures. Starting at room temperature, the Polyguard membrane appears to be the strongest membrane. The word "appears" is used because the membrane could not be pulled apart. During the testing the Polyguard membrane continually slipped out of the grips, even when folded around a steel dowel and reclamped into the grips. However, based on the results from the other temperatures, when the membranes broke, we estimate Polyguard's room temperature tensile strength to be approximately 200 lbf/in. (35,000 N/m). As mentioned, each membrane increased in strength at lower temperatures. Royston, the weakest at room temperature, more than doubled in tensile strength when tested at

Figure 5. Dogbone tensile specimens, ASTM D 2523.
membrane, which only stretched 2% before failing. Thus, all of the membranes, except Royston, showed about equal ability to accommodate movement. We cannot comment as to whether these membranes are sufficient to accommodate the movement of an active crack on a bridge.

**PUNCTURE**

The primary purpose of a bridge membrane is defeated if the membranes are punctured by any means. Quality control during manufacture, transport and installation can help to prevent puncturing the membrane in new decks. Once the membrane is laid, new threats commonly occur. Debris

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**Table 3. Tensile test results.**

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Tensile strength at failure (lb/in.)</th>
<th>Elongation at failure (%)</th>
<th>Test temperature (°F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>W.R. Grace</td>
<td>73.4</td>
<td>14</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>101.4</td>
<td>15</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td>111.0</td>
<td>15</td>
<td>23</td>
</tr>
<tr>
<td></td>
<td>112.3</td>
<td>15</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>122.5</td>
<td>18</td>
<td>-4</td>
</tr>
<tr>
<td>Soprema</td>
<td>138.4</td>
<td>46</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>152.7</td>
<td>33</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td>173.1</td>
<td>36</td>
<td>23</td>
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<td></td>
<td>175.0</td>
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</tr>
<tr>
<td></td>
<td>179.4</td>
<td>14</td>
<td>-4</td>
</tr>
<tr>
<td>Polyguard</td>
<td>111.8*</td>
<td>21*</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>200.3</td>
<td>12</td>
<td>40</td>
</tr>
<tr>
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<td>214.6</td>
<td>12</td>
<td>23</td>
</tr>
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<td></td>
<td>220.7</td>
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<tr>
<td></td>
<td>239.8</td>
<td>12</td>
<td>-4</td>
</tr>
<tr>
<td>Protecto Wrap</td>
<td>64.4</td>
<td>12</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>65.4</td>
<td>14</td>
<td>40</td>
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<td></td>
<td>74.3</td>
<td>14</td>
<td>23</td>
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<td></td>
<td>81.3</td>
<td>14</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>108.9</td>
<td>14</td>
<td>-4</td>
</tr>
<tr>
<td>Royston</td>
<td>60.0</td>
<td>2</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>74.7</td>
<td>2</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td>89.1</td>
<td>2</td>
<td>23</td>
</tr>
<tr>
<td></td>
<td>97.8</td>
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<td></td>
<td>121.32</td>
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<td>NEI</td>
<td>92.5</td>
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<td>107.4</td>
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<tr>
<td></td>
<td>115.8</td>
<td>15</td>
<td>-4</td>
</tr>
</tbody>
</table>

*Membrane pulled out of grips; it did not break. Values represent the average of up to three tension tests.*

---

Figure 6. Tension test setup.

-4°F (-20°C). The others strength increased anywhere from a third to two-thirds. Of all the membranes, the Polyguard membrane provided the most resistance against splitting at all temperatures.

Table 3 also shows how well a membrane can accommodate movement. At room temperature, the Soprema membrane stands out with its ability to stretch 46% before rupturing compared to less than 20% for all the other membranes. However, this room-temperature advantage quickly diminished with each drop in temperature. At -4°F, the Soprema membrane was no better than the others. The only exception to this was the Royston
from neighboring operations can fall on it. Sharp tools may be rested or dropped on it. Some equipment and workers may traffic on it. Next, hot asphalt concrete is laid and vigorously compacted. Over 85% of the volume of a typical asphalt concrete is composed of rock aggregates that have varying degrees of angularity and sharpness. Virtually all aggregates are harder than the membrane material. In hot asphalt concrete, the aggregates are held in place by the cohesion of viscous bitumen binder. The potential for membrane puncture is significant. Therefore, it is desirable that the sheet membrane be able to resist puncture as much as possible. However, the properties of sheet membrane materials vary among sources. Furthermore, the specifications reported by manufacturers are developed by differing test methods. It is nearly impossible for a project engineer to compare sheet membranes by the values reported by different manufacturers as the puncture resistance of their products.

Several standard test methods for puncture resistance evaluation are in use. Perhaps the most frequently used puncture standard test method is ASTM E-154. In this method, the membrane specimen is punctured while supported by a frame, much like in an acoustic drum. However, in a field bridge deck, the membrane is almost continually supported by the deck. In view of the lack of consensus among manufacturers about a puncture test standard, coupled with the unreal drum frame specimen support, the research team decided to devise and propose a new test method.

New puncture test apparatus

The new test method is relatively easy and inexpensive to implement. The test apparatus is commercially available from various sources such as Soiltest, Inc., and Gilson Company, Inc. It is marketed under the name "Acme Laboratory Penetrometer," Soiltest catalog number CT-426 and Gilson catalog number HM-570. The apparatus was originally designed to meet the requirements of ASTM C403 and AASHTO T 197, which measure the rate of hardening of mortar sieved from concrete mixtures, i.e., mortar setting times. This apparatus (Fig. 7) is fairly common in material testing laboratories. The apparatus is portable, does not require electricity to operate, and it weighs about 25 lb (11.4 kg). In addition to the commercially available apparatus, a special, but simple, metal tip must be made for this new test method. The tip can be made of common carbon steel, and it must conform to the geometry shown in Figure 8 below. The tip must be shaped to an angle of 105° with a flat pinnacle of 0.5 mm. The conic tip must terminate in a cylindrical section of about 7 mm in diameter by about 5 mm in length. The cylindrical section that is inserted into the penetrometer must be 12.7 mm (0.50 in.) in diameter by about 50 mm in length (approximately 2 in.). The tip must be free of striations and preferably polished with sand paper with grit of 100 or finer.

The membrane specimen is cut to a manageable size. The size of a standard letter paper (8.5 × 11 in. [21.6 × 27.9 cm]) is suggested. If the membrane uses a sheet of paper to protect its sticky side, this sheet must be removed and replaced by a sheet of aluminum foil. This ensures that the puncture values are not affected by the properties of the temporary backing sheet. At this stage, the specimen is laid on a 6-mm- (about 0.25-in.-) thick steel plate. This steel plate may be approximately the size of half of a standard letter paper (6.5 × 8.5 in. [16.5 × 21.6 cm]). An electrically insulating layer is required to separate the steel plate from the metal base of the apparatus. This insulating layer may be implemented by a letter-size card stock or similar material (8.5 × 11 in. [21.6 × 27.9 cm]).

To detect when a puncture occurs, an ohmmeter is utilized by placing one of its electrodes pinched between the loose steel plate and the insulating layer, and the other held in contact with the metal body of the penetrometer. This way, the ohmmeter will initially indicate an open circuit and it will suddenly change the display value (magnitude is irrelevant) when the circuit is bridged by the contact of the puncture tip with the specimen's conductive substrate. This event instructs the operator to immediately release the puncture load. Figure 9 shows a schematic of the puncture failure detection system. The load is applied at a rate of approximately 20 lb (9.1 kg) per second. The Acme Penetrometer is equipped with an arm that holds the maximum load that was applied during the test. This load is then recorded as the puncture load for one instance of the test. A minimum of 20 instances of puncture tests should be conducted for each membrane sample. The test result is reported as the average puncture load for the number of instances of the puncture test conducted for the membrane being evaluated. In addition, the coefficient of variation* for the test set should be reported.

*The coefficient of variation in a set of tests is calculated by dividing the standard deviation by the average and then multiplying this ratio by 100.
Figure 7. Commercially available instrument used as the basis for the proposed apparatus.

Figure 8. Tip geometry.
The room temperature during the test must be kept approximately constant. It is recommended that the air temperature during testing be kept at about 20°C ± 5°C. The temperature of the environment, the specimen, and the test apparatus of the proposed test method were designed for ease of implementation. This avoids the need for heaters or refrigeration. It is acknowledged that the real field puncture temperature will normally be higher. Evaluating membranes for their resistance to puncture at temperatures representative of field conditions may be another good approach to evaluating membranes, but it would increase the complexity of the test method. This later approach falls outside the scope of the current research and development effort, but it may be a valuable component of future work.

Test results
To use a commercially available instrument to perform the proposed puncture tests, a number of preliminary tests were conducted. The critical parameter for this calibration was the tip angle. A very acute tip angle rendered the test results clustered at the lower sector of the valid range of measurement of the apparatus. A very obtuse angle caused many of the tests to fall outside the upper measurement limit of the apparatus. Figure 10 shows the test results of a set of 20 puncture tests for each of the six membranes under evaluation. The figure illustrates how some of the values exceeded the 200-lb (91-kg) measurement range of the apparatus. These values corresponded to a tip angle of 110°.

Figures 11 through 13 show the test results of three separate sets of 20 tests for each of the six sheet membranes under study. These figures demonstrate that the proposed test method is effective at rating membranes by their resistance to puncture. Some of the dispersion of the test results is inherent of the test methodology, and some indicate the degree of consistency found throughout the specimens from a given source. Therefore, some membranes clearly have superior puncture resistance than others, but in some cases, the differences were not clear because their magnitude was comparable to the magnitude of the data dispersion.

Figures 11 through 13 show that the test results were mostly consistent across the different test sets. Appendix D shows the test results for each test set in tabular format.

Statistical significance of the test results
Table 4 shows statistical results derived from the three test sets presented in Figures 11 through 13. The test results were ordered from most resistant to least resistant according to the average of all three test sets. The coefficient of variation is the most significant statistical parameter applicable to this case. It indicates the magnitude of the scattering of the data expressed in a format that allows comparison across the various sheet membranes. The coefficient of variation allows comparison that is scaled to the magnitude of the average values of the test results. The data indicate that, out of the six membranes evaluated, the Soprema specimens have clearly superior puncture resistance. Soprema’s lowest coefficient of variation also indicates superior consistence from specimen to specimen. The puncture resistance of the various sheet membranes was influenced by a combination of their properties such as thickness, surface layer material, and properties of the base membrane material. The bridge designer must consider
Figure 10. Puncture test results during the determination of the tip angle.

Figure 11. Puncture test results for first test set.
these and other factors in the process of selecting sheet membranes.

The test results indicate that the NEI and the Polyguard sheet membranes ranked second and third in this evaluation. However, the significance of their relative resistance is obscured by the magnitude of the data scattering. The significance of the superiority of these two membranes with respect to the bottom three membranes can be asserted because it exceeds the magnitude of the data scattering, i.e., the standard deviation. The test data do not allow a statistically valid distinction.
Table 4. Statistical analysis of puncture test results.

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Average</th>
<th>Standard deviation</th>
<th>Coefficient of variation</th>
</tr>
</thead>
<tbody>
<tr>
<td>First set of 20 tests</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Soprema</td>
<td>157.7</td>
<td>13.6</td>
<td>8.6</td>
</tr>
<tr>
<td>NEI</td>
<td>88.4</td>
<td>9.3</td>
<td>10.5</td>
</tr>
<tr>
<td>Polyguard</td>
<td>71.6</td>
<td>10.6</td>
<td>14.8</td>
</tr>
<tr>
<td>Royston</td>
<td>50.3</td>
<td>4.0</td>
<td>7.9</td>
</tr>
<tr>
<td>Protecto Wrap</td>
<td>38.5</td>
<td>4.1</td>
<td>10.7</td>
</tr>
<tr>
<td>W.R. Grace</td>
<td>36.5</td>
<td>6.7</td>
<td>18.3</td>
</tr>
<tr>
<td>Column average</td>
<td>73.8</td>
<td>8.0</td>
<td>11.8</td>
</tr>
<tr>
<td>Second set of 20 tests</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Soprema</td>
<td>148.5</td>
<td>12.3</td>
<td>8.3</td>
</tr>
<tr>
<td>NEI</td>
<td>99.5</td>
<td>16.3</td>
<td>16.4</td>
</tr>
<tr>
<td>Polyguard</td>
<td>53.8</td>
<td>7.0</td>
<td>13.0</td>
</tr>
<tr>
<td>Royston</td>
<td>51.7</td>
<td>10.0</td>
<td>19.5</td>
</tr>
<tr>
<td>Protecto Wrap</td>
<td>39.7</td>
<td>3.4</td>
<td>8.6</td>
</tr>
<tr>
<td>W.R. Grace</td>
<td>36.0</td>
<td>6.3</td>
<td>17.5</td>
</tr>
<tr>
<td>Column average</td>
<td>71.5</td>
<td>9.2</td>
<td>13.8</td>
</tr>
<tr>
<td>Third set of 20 tests</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Soprema</td>
<td>142.6</td>
<td>16.1</td>
<td>11.3</td>
</tr>
<tr>
<td>NEI</td>
<td>90.9</td>
<td>10.4</td>
<td>11.4</td>
</tr>
<tr>
<td>Polyguard</td>
<td>70.0</td>
<td>12.5</td>
<td>17.8</td>
</tr>
<tr>
<td>Royston</td>
<td>42.1</td>
<td>4.4</td>
<td>10.5</td>
</tr>
<tr>
<td>Protecto Wrap</td>
<td>37.5</td>
<td>7.1</td>
<td>19.0</td>
</tr>
<tr>
<td>W.R. Grace</td>
<td>39.0</td>
<td>8.1</td>
<td>20.7</td>
</tr>
<tr>
<td>Column average</td>
<td>70.3</td>
<td>9.8</td>
<td>15.1</td>
</tr>
<tr>
<td>Fourth set of 20 tests</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Soprema</td>
<td>149.6</td>
<td>14.0</td>
<td>9.4</td>
</tr>
<tr>
<td>NEI</td>
<td>92.9</td>
<td>12.0</td>
<td>12.9</td>
</tr>
<tr>
<td>Polyguard</td>
<td>65.1</td>
<td>10.0</td>
<td>15.4</td>
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<tr>
<td>Royston</td>
<td>48.0</td>
<td>6.1</td>
<td>12.7</td>
</tr>
<tr>
<td>Protecto Wrap</td>
<td>38.5</td>
<td>4.9</td>
<td>12.6</td>
</tr>
<tr>
<td>W.R. Grace</td>
<td>37.2</td>
<td>7.0</td>
<td>18.9</td>
</tr>
<tr>
<td>Column average</td>
<td>71.9</td>
<td>9.0</td>
<td>13.7</td>
</tr>
</tbody>
</table>

among the bottom three membranes, because the magnitudes of their data scattering exceed the differences in their puncture resistance.

WATER VAPOR PERMEANCE

This study reviewed and evaluated the suitability of the ASTM E96-95 Standard Test Methods for Water Vapor Transmission of Materials to sheet membranes used as waterproofing between concrete bridge decks and asphalt overlays used as the trafficking surface. A modified test method, which is under consideration for inclusion in ASTM E96, was investigated and the results compared to a variation of one of the standard test methods.

The test methods in ASTM E96 cover the determination of water vapor transmission (WVT) of materials through which the passage of water vapor may be of importance. There are two basic methods, the Desiccant Method and the Water Method. These methods are provided for the measurement of permeance, and two variations include service conditions with one side wetted and service conditions with low humidity on one side and high humidity on the other. Some suggested standard test conditions from E96 are listed below:

Procedure A – Desiccant Method at 73.4°F (23°C)
Procedure B – Water Method at 73.4°F (23°C)
Procedure BW – Inverted Water Method at 73.4°F (23°C)
Procedure C – Desiccant Method at 90°F (32.2°C)
Procedure D – Water Method at 90°F (32.2°C)
Procedure E – Desiccant Method at 100°F (37.8°C) and a 90% RH

Procedures A through D are normally conducted in a test chamber at 50% relative humidity. Agreement should not be expected between results obtained by different methods and that method should be selected which more nearly approaches the conditions of use.

A "perm" is the inch-pound unit of measurement for permeance. One "perm" is the mass rate of water vapor flow through one square foot of material or construction of one grain (gr) per hour induced by a vapor pressure gradient between two surfaces of one inch of mercury or in units that equal that flow rate (JTI 1990):

\[
1 \text{ perm} = 1 \text{ gr/h} \cdot \text{ft}^2 \cdot \text{in. Hg} = 5.75 \times 10^{-11} \text{ kg/s} \cdot \text{m}^2 \cdot \text{Pa}
\]

Historically, a material or system with a permeance of 1 perm or less qualifies as a vapor retarder. More recently, further classification of vapor retarders has been proposed. For example, the Canadian General Standards Board (CGSB) has specified Type I vapor retarders as ones with a permeance of 0.25 perm or less, and Type II as retarders with a permeance of 0.75 perm or less before aging and 1 perm or less after aging (ASHRAE 1997).

Table 5 summarizes the membrane manufacturers' data for the six membranes tested. The data are sorted from lowest to highest permeance and shows that these values range from 0.003 to 1 perm and vary by a factor of 333. The membrane thickness varies from 60 to 170 mils. The manufacturers generally tested their membranes using E96
Method B; however, two manufacturers did not specify the method, only that E96 was the standard used. Since agreement should not be expected between results obtained by different methods, it makes sense to specify a method and require all membranes to be tested accordingly.

There are several critical aspects of the ASTM E96 procedure that can adversely affect the permeance results. Toas (1989) reported on a 1985 round-robin test series using the E96 procedure. He stated that in performing the ASTM E96 test, test operators must seal the test sample perfectly to the test dish, carefully weigh the sealed specimen with a balance having the proper sensitivity, and maintain the proper atmosphere for the test. Low permeance materials are difficult to measure, as the weight gain or loss is usually quite small and the potential for error is much greater. Consequently, three material specimens are required, and a "dummy" should be used when testing materials with a permeance less than 0.05 perm. Statistically significant results require at least four repetitions of each membrane. These results usually are determined using a least squares regression analysis of the weight loss or gain, modified by the dummy specimen as a function of time to obtain the water vapor transmission. We did not attempt a statistical study as this would have been too costly. We decided instead to proof test an alternative method and compare it to a variation of a standard method.

**Procedures**

We investigated a "Modified-Cup Method" as a result of our review and conversations with others familiar with the E96 test procedures. Figure 14 shows a schematic of the modified-cup apparatus for water vapor permeance testing. This method combines the features of both the ASTM dry and wet cup methods (Schwartz et al. 1989). The modified test method creates the maximum vapor pressure difference possible at the test temperature of 84°F (29°C) that we used. This modified method eliminates the need to maintain a 50% RH environment on one side of the sample. That saves a lot of effort in setting up and maintaining a fixed relative humidity in the test chamber. Figure 15 shows the modified-cup apparatus used for permeance testing. The apparatus is held tightly together by two aluminum plates bolted together. The assembly is placed in the test chamber and periodically disassembled so that the water cup, specimen, and desiccant cup portions can be weighed. Then it is reassembled and the test continued.

We compared the results of the modified-cup to a variation of the standard water test method using a test temperature of 84°F (rather than 73.4°F [23°C]) and an RH of 16% (rather than 50%). We also poked a hole through three of the membranes and re-tested these membranes using the water-cup method.

**Sample preparation**

Figure 16 shows a membrane sample ready for testing. The membrane samples were adhered to a stainless steel mounting plate using two techniques. The first technique used pressure-sensitive adhesives on five of the six membranes tested. A mounting jig was used to center these membranes on their mask, and then the steel plate was pressed onto the membrane (after the release sheet

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Product tested</th>
<th>Thickness (mils)</th>
<th>ASTM test method</th>
<th>Permeance (perm)</th>
<th>Permeance (g/s · m² · Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Protecto Wrap</td>
<td>M140A-R</td>
<td>60</td>
<td>E96 Method B</td>
<td>0.003</td>
<td>1.7x10^-10</td>
</tr>
<tr>
<td>Soprema</td>
<td>Soralene Flam Antitrol</td>
<td>170</td>
<td>E96 Method B</td>
<td>0.0036</td>
<td>2.1x10^-10</td>
</tr>
<tr>
<td>Royston</td>
<td>10AN Easy Pave ER</td>
<td>60</td>
<td>E96 Method B</td>
<td>0.05</td>
<td>2.9x10^-9</td>
</tr>
<tr>
<td>NEI</td>
<td>AC Bridge and Deck Seal</td>
<td>65</td>
<td>E96 Method B</td>
<td>0.08</td>
<td>4.6x10^-9</td>
</tr>
<tr>
<td>Polyguard</td>
<td>665 LT</td>
<td>65</td>
<td>E96 Method B</td>
<td>0.1</td>
<td>5.7x10^-9</td>
</tr>
<tr>
<td>W.R. Grace</td>
<td>Bituthene 5000</td>
<td>65</td>
<td>E96 Method B</td>
<td>1</td>
<td>5.8x10^-8</td>
</tr>
</tbody>
</table>
Figure 14. Schematic shows apparatus for water vapor permeance tests.

Figure 15. Apparatus for water vapor permeance test (Modified Cup Method).

Figure 16. Sample ready for water vapor permeance testing.
had been removed). The material was rolled with
a steel roller to increase the adhesion of the mem-
brane to the plate. The other technique used heat
to seal the modified bitumen membrane to the
plate. Again the sample was centered on the mask
in the mounting jig, the sample back was heated
with a propane torch until the asphalt started to
melt, and then the steel plate was pressed down
onto the sample.

Once the bottom of a membrane was adhered
to the mounting plate, the top of the sample was
coated with wax around its perimeter to a width
equal to that of the mask. The mask was then
heated for a several seconds and applied to the
wax on the sample. Finally the edge of the sample
and mask was sealed with wax applied on in mul-
tiple layers. The sample application procedure
requires practice to become familiar with all the
possible difficulties that can occur during the pro-
cedure.

Each membrane sample was used for two per-
meance tests. The first test was the modified-cup
test with the desiccant above the sample and the
water below as shown in Figure 14. The second
water-cup test used only the water cup fastened
to the sample plate. Three membranes with a 0.06-
in.-diam. hole punched through them with a
heated steel rod were also retested using the wa-
ter-cup method.

Test chamber and controls
An insulated walk-in box was used as the test
chamber. A proportionally controlled electric
heater maintained the temperature in the cham-
ber. That temperature was kept at 84°F plus or
minus about a degree. An open pan of water main-
tained the relative humidity of the chamber at
about 16%, plus or minus about 2%.

Measurement problems
At the beginning of the tests, we discovered that
there was a significant variation in successive
weights of individual desiccant and water cups
due to static electricity created by handling of the
cups. We finally solved this problem by isolating
the sample far enough from the test scale with a
piece of wood, so that the attraction between the
scale’s case and the cups due to the static elec-
tricity was minimized.

When we began making test weights in the
modified test procedure, we discovered that there
was a significant amount of weight change while
we were making and recording the weights (about
the same magnitude as the weight changes be-
tween measurements). This required an adjust-
ment of the procedure to minimize that weight
loss/gain. We were able to tare out those losses
and gains.

Water losses and desiccant gains are to be ex-
pected when the cup is opened for weighing.
Losses and gains can occur through the closed
seals of the apparatus. We suspect that our seals
leaked as, in hindsight, we used an inappropriate
material (nylon) for the threaded rods (shown at
the corners of the cup assembly in Figure 15).
Because of this choice, we could not apply as much
force to clamp the assembly together as should
have been used. Thus the moisture could leak in
or out through the machining grooves at the bot-
tom of the O-ring slot, along the surface of the
mating cup, or through scratches along the sur-
face of the sample plate ring.

Test results
Table 6 and Appendix E show the modified-cup
test results. The y-axis range on both sides of all
six graphs in Appendix E is 5 grains (since 1 grain
= 15.432 grains, the y-axis range is only about a
third of a gram). There are two measurements of
water vapor transmission and permeance for each
membrane in the modified-cup test method. One
is the weight gain by the desiccant; the other is
the weight loss from the water. If there were no
leaks and the membrane absorbed no moisture
then the weight gain would equal the weight loss.
We suspect that our apparatus leaked or that our
ability to tare out losses or gains was not adequate.
Consequently, we used the smaller of the weight
gain of the desiccant or the weight loss of the wa-
ter to calculate the weight change per hour for the
material, since that smaller value has the lower
extraneous loss or gain included. For low per-
meance materials, ASTM E96 specifies that the
reported permeance is the linear regression slope
of the weight change vs. time divided by the
sample area and the average pressure difference.
The third column of data in Table 6 shows the
weight change per hour used to calculate the per-
meance (linear regression slope of the lines in
App. E). Dividing the weight change per hour
by the sample area yields the water vapor trans-
mission (WVT). Dividing the WVT by the pres-
sure difference across the sample yields the per-
meance. Table 7 and Appendix F show the more
conventional water-cup test results. The y-axis
range of all six graphs in Appendix F is 5 grains.
Again the slope method or "numerical analysis"
was used to determine the weight change per hour
of the water cup (linear regression slope of the lines in App. F). WVT and permeance were calculated as discussed above. All weight changes in Tables 6 and 7 are corrected for changes in barometric pressure and due to measurement procedures in the case of Table 6.

The results of both these tests show that the membranes have a permeance of about 0.01 to 0.05 perm. The results of these tests show that the permeance of all six tested membranes is nearly the same. The average permeance for the modified-cup procedure is 0.030 perm, and the average value for the water method is 0.028 perm. Both tests were run at an average temperature of 84°F.

Table 8 compares both of our permeance test results to the manufacturers' data. Note that test results from our water-cup method are lower than those of our modified-cup method in all cases. We attribute the decrease to problems experienced in adequately tightening the modified-cup to avoid leakage through the seals. Four manufacturers provided permeance test reports just before we concluded our testing. Permeance values from these reports are italicized in Table 8 just below the original published values. We consider the extremely low permeance report value for the NEI membrane to be suspect. Our water-cup test permeance values are also lower than those reported by all six manufacturers (considering their report data and ignoring the suspect NEI value). All six membranes are certainly water vapor resistant.

Three membranes were retested using the water-cup method after they were punctured with a heated 0.06-in.-diam. steel rod. The permeance of the least permeable membrane (Soprema) increased by an order of magnitude when we added the hole. The permeance of the next more permeable membrane (NEI) increased by a factor of 7 when we added the hole. And the permeance of the most permeable membrane (Royston) increased by a factor of 1.3 when we added the hole.

These tests are difficult to conduct, and errors may be introduced during sample preparation, sample handling, and when obtaining weights and

Table 6. Modified cup method.

<table>
<thead>
<tr>
<th>Membrane manufacturer</th>
<th>Dry cup weight gain (gr)</th>
<th>Wet cup weight loss (gr)</th>
<th>Wt. change per hour (gr/hr)</th>
<th>WVT (gr/hr - ft²)</th>
<th>Permeance (perm) or (gr/hr - ft² - in. Hg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NEI</td>
<td>1.888</td>
<td>2.099</td>
<td>0.0037</td>
<td>0.038</td>
<td>0.034</td>
</tr>
<tr>
<td>Polyguard</td>
<td>2.269</td>
<td>1.651</td>
<td>0.0033</td>
<td>0.034</td>
<td>0.030</td>
</tr>
<tr>
<td>Protec Wrap</td>
<td>2.222</td>
<td>1.511</td>
<td>0.0029</td>
<td>0.029</td>
<td>0.026</td>
</tr>
<tr>
<td>Royston</td>
<td>3.272</td>
<td>2.498</td>
<td>0.0058</td>
<td>0.059</td>
<td>0.052</td>
</tr>
<tr>
<td>Soprema</td>
<td>0.741</td>
<td>1.173</td>
<td>0.0014</td>
<td>0.014</td>
<td>0.012</td>
</tr>
<tr>
<td>W.R. Grace</td>
<td>3.177</td>
<td>1.589</td>
<td>0.0031</td>
<td>0.031</td>
<td>0.027</td>
</tr>
</tbody>
</table>

*Using the smaller of the weight gain of the desiccant or the weight loss of the water per hour by the slope method as described in ASTM E 96.
Test temperature was 84°F for an average vapor pressure difference of 1.1316 in. of Hg.
The sample area in all cases was 0.0985 ft² and the length of the test was 560 hours.

Table 7. Wet-cup or water method with the following test conditions.

<table>
<thead>
<tr>
<th>Membrane manufacturer</th>
<th>Total weight loss (gr)</th>
<th>Weight loss per hour (gr/hr)</th>
<th>WVT (gr/hr - ft²)</th>
<th>Permeance (perm) or (gr/hr - ft² - in. Hg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NEI</td>
<td>1.914</td>
<td>0.0025</td>
<td>0.025</td>
<td>0.025</td>
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<tr>
<td>Polyguard</td>
<td>2.022</td>
<td>0.0026</td>
<td>0.026</td>
<td>0.026</td>
</tr>
<tr>
<td>Protec Wrap</td>
<td>1.790</td>
<td>0.0021</td>
<td>0.021</td>
<td>0.021</td>
</tr>
<tr>
<td>Royston</td>
<td>3.596</td>
<td>0.0048</td>
<td>0.049</td>
<td>0.050</td>
</tr>
<tr>
<td>Soprema</td>
<td>0.849</td>
<td>0.0011</td>
<td>0.011</td>
<td>0.011</td>
</tr>
<tr>
<td>W.R. Grace</td>
<td>2.330</td>
<td>0.0031</td>
<td>0.031</td>
<td>0.032</td>
</tr>
</tbody>
</table>

*Using the slope method as described in ASTM E 96.
Test conditions were 84°F and 16% RH for an average vapor pressure difference of 0.9824 in. of Hg.
The sample area in all cases was 0.0985 ft² and the length of the test was 720 hours.
Table 8. Permeance test results compared to manufacturers’ data, sorted by lowest to highest permeance as published by the manufacturers.

<table>
<thead>
<tr>
<th>Membrane manufacturer</th>
<th>Modified cup method permeance (perm) or (gr/hr-ft²-in. Hg)</th>
<th>Wet cup (perm) or (gr/hr-ft²-in. Hg)</th>
<th>Wt. change per hour* (gr/hr-ft²-in. Hg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Protecto Wrap</td>
<td>0.026</td>
<td>0.003</td>
<td>0.021</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.049</td>
<td></td>
</tr>
<tr>
<td>Soprema</td>
<td>0.012</td>
<td>0.0036</td>
<td>0.011</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.02</td>
<td></td>
</tr>
<tr>
<td>Royston</td>
<td>0.052</td>
<td>0.05</td>
<td>0.050</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.08</td>
<td>0.025</td>
</tr>
<tr>
<td>NEI</td>
<td>0.034</td>
<td>0.000644</td>
<td></td>
</tr>
<tr>
<td>Polyguard</td>
<td>0.030</td>
<td>0.1</td>
<td>0.026</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.035</td>
<td></td>
</tr>
<tr>
<td>W.R. Grace</td>
<td>0.027</td>
<td>1</td>
<td>0.021</td>
</tr>
</tbody>
</table>

*The permeance values shown in the top row are those found in the manufacturers’ data at the beginning of our testing. The italicized permeance values shown in the second row were provided later by the manufacturer.

calculating the results. We also had difficulty in properly maintaining test conditions throughout the test. There may also be problems with unit conversions, making it difficult to compare results from different laboratories and manufacturers.

At the beginning of these tests, we wondered if blisters would be less likely to develop if a membrane can pass water vapor and air but not allow liquid water to pass. However, these water vapor transmission tests do not allow us to determine if that is possible. These membranes are effective water vapor retarders and are not likely to act as a ventilating layer.

Neither procedure (modified-cup or wet-cup) tests the membrane in the condition of use (i.e., sandwiched between two permeable materials that are capable of puncturing the membrane). Such a method would require much more development time and numerous trials before common acceptance would be likely.

CONCLUSIONS

Adhesion
- Poor adhesion is the primary cause of blistering.
- A membrane does not have to be perfectly adhered to the deck to avoid blistering.
- High bond strength matters less than the continuity of the bond.
- A blister can originate in a smaller void containing water than one containing dry air.
- Bridge inspectors should be alerted to the fact that a membrane may not be adequately installed if it has unadhered areas larger than a quarter coin. This is the smallest void size that can originate a blister.
- Once a blister initiates, even though it may not immediately be evident, it will eventually become a problem.
- The Protecto Wrap and W.R. Grace membranes showed the least tendency to contain weak adhesion spots.
- The Protecto Wrap membrane’s bond was the one least affected by membrane applicator.

Tensile strength and elongation
- Membranes are most prone to splitting during cold weather when cracks in the concrete deck are at their widest.
- While strength and elongation both describe tensile properties, the latter is considered most appropriate to judge a membrane’s ability to span a crack.
- The Soprema membrane had the best elongation at room temperature.
- The Royston membrane was the least stretchable at all temperatures.
- At -4°F, all membranes, except for Royston’s, were considered comparable in their ability to span an active crack.
Puncture resistance

- Puncture of a bridge membrane defeats the purpose of its existence.
- The potential for puncture is significant in the bridge deck construction environment.
- Puncture resistance is an important property of a good sheet membrane.
- The lack of consensus on a test standard for the evaluation of sheet membranes indicates the need for a uniform methodology for puncture resistance evaluations.
- The proposed test method more closely approximates field conditions. The test apparatus is relatively inexpensive, commercially available, and portable. Specimen preparation is relatively simple.
- The puncture test results obtained during this evaluation show that the proposed test method is adequate to rate membranes according to their puncture resistance.
- The bridge designer must consider the results from the various test types along with other factors relevant to the selection of sheet membranes, such as cost, environmental friendliness, and durability.

Water vapor permeability

- All six membranes are very good vapor retarders. Their permeance ranged from 0.01 to 0.05 perm and varied by a factor of five, instead of the factor of 333 originally indicated in the manufacturers’ data.
- The modified-cup test procedure has potential, but our apparatus needs some refinement. This procedure provides both a water weight loss and a desiccant weight gain across a specimen without the need for humidity control. The weight loss should equal the weight gain if leakage is controlled and the sample absorbs no moisture.
- Manufacturers seem to prefer the ASTM E96 (Water-Cup Method B) and we see no reason to change that. However, all bridge membranes should be subjected to the same test method and test conditions if reliable comparisons are to be made between them. It would be best if all testing were done by one highly qualified lab that is aware of the potential for errors when using ASTM E96.

RECOMMENDATIONS

Until a membrane has been field tested, its service life remains unknown. Laboratory tests can help rank membranes according to individual properties, but exposure to the complex combination of natural forces is essential for proving a material’s durability. Therefore, it is recommended that a field study be instituted to monitor the installation/design process and long-term performance of bridge membranes in New England.

Sheet membranes are one type of waterproofing—liquid applied membranes are the other. It is recommended that a parallel test be conducted on liquid membranes to complete the picture.

LITERATURE CITED


Hironaka, M. C., and T. J. Holland (1986) Blistering of asphalt pavement overlay on runway 14-32 at MCAS Beaufort, South Carolina. Naval Civil Engineering Laboratory, Port Hueneme, California, NCEL Technical Note 1744.
### APPENDIX A: MANUFACTURERS' DATA FROM PRODUCT BROCHURES FOR REFERENCE PURPOSES

<table>
<thead>
<tr>
<th>Membrane manufacturer</th>
<th>Soprema</th>
<th>Polyguard</th>
<th>Protecto Wrap</th>
<th>W.R. Grace</th>
<th>Royecon</th>
<th>NEI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Sopralene Flam Antirock</td>
<td>665 LT</td>
<td>M140A-R</td>
<td>Bituthene 5000</td>
<td>10AN Easy Pave ER</td>
<td>AC Bridge &amp; Deck Seal</td>
</tr>
<tr>
<td>Primer</td>
<td>Elastoco 500</td>
<td>650 RC</td>
<td>80 Primer</td>
<td>P 3000 Primer</td>
<td>Roybond 713A Primer</td>
<td>AC Primer</td>
</tr>
<tr>
<td>Thickness of membrane</td>
<td>0.170 in.</td>
<td>0.065 in.</td>
<td>0.060 in.</td>
<td>0.065 in.</td>
<td>0.060 in.</td>
<td>0.065 in.</td>
</tr>
<tr>
<td>Reinforcement</td>
<td>nonwoven polyester</td>
<td>polypropylene mesh (at the top surface)</td>
<td>polyester, stitch-bonded</td>
<td>0.005 in. polypropylene (at the top surface)</td>
<td>nonwoven fiberglass</td>
<td>nonwoven polyester</td>
</tr>
<tr>
<td>Tensile strength (*= reinforcement only)</td>
<td>90 lbf/in.</td>
<td>200 lbf/in.</td>
<td>24 lbf/in.* (0.015 in. thick)</td>
<td>50 lbf/in.</td>
<td>25 lbf/in.</td>
<td>85 lbf/in.</td>
</tr>
<tr>
<td>Test method</td>
<td>CGSB 37GP56M; ASTM D 882 (Method B)</td>
<td>ASTM D 882 (Method A)</td>
<td>ASTM D 882</td>
<td>ASTM D 882</td>
<td>ASTM D 1000 (Mod.)</td>
<td>ASTM D 412</td>
</tr>
<tr>
<td>Tensile tear strength (lbf)</td>
<td>155</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>ASTM D 4703</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Elongation (%)</td>
<td>8</td>
<td>500 min., “ultimate failure of tear mass”</td>
<td>25 (mesh)</td>
<td>1600 (compound)</td>
<td>300 (compound)</td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>CGSB 37GP56M</td>
<td>ASTM D 882 (Method A)</td>
<td>ASTM D882</td>
<td>ASTM D1000 (Mod.)</td>
<td>ASTM D412</td>
<td></td>
</tr>
<tr>
<td>Permeance, perms (grains/ft²/hr in. Hg)</td>
<td>0.0036</td>
<td>0.1</td>
<td>0.003</td>
<td>1</td>
<td>0.05</td>
<td>0.08</td>
</tr>
<tr>
<td>Test method</td>
<td>ASTM E-96 (Method B)</td>
<td>ASTM E 96 (Method B)</td>
<td>ASTM E96 (Method B)</td>
<td>ASTM E96 (Method B)</td>
<td>ASTM E96</td>
<td></td>
</tr>
<tr>
<td>Water absorption, %</td>
<td>0.6</td>
<td>0.25</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>ASTM D 5147</td>
<td>ASTM D1288 (@ 72 hr)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Resistance to hydrostatic head</td>
<td>150 ft</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>RTM 29f</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Puncture resistance (lbf)</td>
<td>245 N</td>
<td>&gt;200</td>
<td>140 (avg.)</td>
<td>200 (mesh)</td>
<td>25</td>
<td>150</td>
</tr>
<tr>
<td>Test method</td>
<td>CGSB 37GP56M</td>
<td>ASTM E154</td>
<td>ASTM E154</td>
<td>ASTM E154</td>
<td>ASTM E154</td>
<td>ASTM E154</td>
</tr>
<tr>
<td>Pliability</td>
<td>&lt;= -35°C</td>
<td>passes, @-15°F</td>
<td>passes</td>
<td>unaffected, @25°F</td>
<td>passes, @-25°F</td>
<td>passes, @-25°F</td>
</tr>
</tbody>
</table>
## APPENDIX A: MANUFACTURERS’ DATA FROM PRODUCT BROCHURES FOR REFERENCE PURPOSES

<table>
<thead>
<tr>
<th>Membrane manufacturer</th>
<th>Soprema</th>
<th>Polyguard</th>
<th>Protecto Wmp</th>
<th>W.R. Grace</th>
<th>Rayston</th>
<th>NEI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Softening point</td>
<td>$\geq 110^\circ$C</td>
<td></td>
<td></td>
<td>$\geq 98^\circ$C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>ASTM D 3686</td>
<td></td>
<td></td>
<td>ASTM D 36</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Penetration</td>
<td>$60-75 \times 77^\circ$F, 5 sec @ 100 g needle</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>ASTM D5</td>
<td></td>
<td></td>
<td>$\text{&quot;unaffected,&quot;}$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Crack cycling</td>
<td>$\text{&quot;unaffected,&quot;}$</td>
<td>@ +25°F through 100 cycles</td>
<td>$\text{&quot;unaffected,&quot;}$</td>
<td>@ 25°F over 1/8-in. crack</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>ASTM C836</td>
<td></td>
<td></td>
<td>ASTM C836</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cycling shear strength (lb/in.²)</td>
<td>25</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>RTM – 301; (2 in./min. @ 32°F, w/ 0 in. opening &amp; 1/8 in. displacement)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cycling shear strength recovery</td>
<td>$\text{&quot;Constant load @ 1000 cycles, no damage&quot;}$</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>RTM – 301; (1/8-in. recovery @ 32°F)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Peel adhesion (180°) (lb/in.)</td>
<td>$\geq 58$</td>
<td>5</td>
<td>25</td>
<td>10, dry</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>ASTM D903</td>
<td></td>
<td>180° peel after 1 hr, primed steel</td>
<td>ASTM D 903</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Compound penetration</td>
<td>$60-75,$</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test method</td>
<td>ASTM D5</td>
<td></td>
<td>(77°F, 5 sec, w/ 100-g needle)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*RTM uses membrane properly applied to two primed steel panels with a 1-in. gap between panels. At a specified temperature the gap is cycled to the specified opening for the specified no. of cycles. For shear strength, the force per unit of the first cycle is recorded. For shear stress recovery, any damage after the no. of cycles to constant load and the no. of cycles required to reach constant load is recorded.

†Hydrostatic head tests are performed on membrane properly applied to primed concrete. The surface is sealed with a pressure chamber and water is introduced under pressure equal to the specified head.
APPENDIX B: ADHESION TEST DATA

The data are presented in groupings of six graphs for each mortar slab. There are a total of 33 such groupings. In each grouping, Figure a is a composite of all data obtained from a particular slab while Figures b through f present individual force-displacement results for each 1-in.-wide strip peeled off that slab. Each strip was peeled off the slabs in two stages. The zero force reading at approximately the 2- to 2.25-in. mark separates the two stages.

By “thumbing” through the data, one quickly becomes aware of the variable nature of adhesion. It fluctuates, sometimes wildly, along the distance of the peel. Clearly, not all membranes are the same. Some grip quite strongly, at least in spots, while others grip more uniformly to the mortar, but not as strongly.
Bituthene by WRG
Supplier Mortar Slab #1

a. Five strips
b. Strip #1
c. Strip #2
d. Strip #3
e. Strip #4
f. Strip #5
Bituthene by WRG
Supplier Mortar Slab #2

a. Five strips
b. Strip #1
c. Strip #2
d. Strip #3
e. Strip #4
f. Strip #5
Bituthene by WRG
Supplier Mortar Slab #3

a. Five strips
b. Strip #1
c. Strip #2
d. Strip #3
e. Strip #4
f. Strip #5
Bituthene by WRG
CRREL Mortar Slab #1

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
Bituthene by WRG
CRREL Mortar Slab #3

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
AC by NEI
CRREL Mortar Slab #1

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
AC by NEI
CRREL Mortar Slab #2

a. Five strips
b. Strip #1
c. Strip #2
d. Strip #3
e. Strip #4
f. Strip #5
AC by NEI
CRREL Mortar Slab #3

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
665LT by Polyguard
Supplier Mortar Slab #1

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
665LT by Polyguard
CRREL Mortar Slab #2

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
665LT by Polyguard
CRREL Mortar Slab #3

Displacement (in.)

Adhesion (lbf/in.)

a. Five strips

b. Strip #1

Malfunction. Data for Strip #3 not recorded.

c. Strip #2

Malfunction. Data for Strip #4 not recorded.

Malfunction. Data for Strip #5 not recorded.
M140AR by Protecto Wrap
Supplier Mortar Slab #1

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
M140AR by Protecto Wrap
Supplier Mortar Slab #2

a. Five strips
b. Strip #1
c. Strip #2
d. Strip #3
e. Strip #4
f. Strip #5
M140AR by Protecto Wrap
Supplier Mortar Slab #3

a. Five strips

b. Strip #1. Data for stage 2 unavailable, malfunction

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
M140AR by Protecto Wrap
CRREL Mortar Slab #1

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
M140AR by Protecto Wrap
CRREL Mortar Slab #3

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
Antirock by Sporema
Supplier Mortar Slab #1

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5

47
Antirock by Sporema
Supplier Mortar Slab #2

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
Antirock by Sporema
Supplier Mortar Slab #3

a. Five strips
b. Strip #1
c. Strip #2
d. Strip #3
e. Strip #4
f. Strip #5
Antrock by Sporema
CRREL Mortar Slab #1

a. Five strips
b. Strip #1
c. Strip #2
d. Strip #3
e. Strip #4
f. Strip #5
Antirock by Sporema
CRREL Mortar Slab #2

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
Antilock by Sporema
CRREL Mortar Slab #3

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
Easy Pave ER by Royston
CRREL Mortar Slab #1

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3. Stage 1 data unavailable, malfunction.

e. Strip #4

f. Strip #5
Easy Pave ER by Royston
CRREL Mortar Slab #2

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
Easy Pave ER by Royston
CRREL Mortar Slab #3

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
Easy Pave ER by Royston
Supplier Mortar Slab #1

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
Easy Pave ER by Royston
Supplier Mortar Slab #2

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
Easy Pave ER by Royston
Supplier Mortar Slab #3

a. Five strips

b. Strip #1

c. Strip #2

d. Strip #3

e. Strip #4

f. Strip #5
APPENDIX C: TENSILE AND ELONGATION TEST DATA

The data are presented in groupings of six graphs, one group per membrane. Each grouping corresponds to a particular test temperature. Elongation was measured using two methods: (1) change in distance between the grips (6.5 in. apart), and (2) change in distance between contact points of an extensometer affixed to the specimen (2.5 in. apart). Each graph consists of two curves where each curve represents the average of three samples: one curve for measurements made with the extensometer compared to those made with the grips.

The elongation measured by movement of the grips is, in nearly all cases, markedly different from that obtained with the extensometer. The explanation is that as the membrane stretches, the gauge length increases as the bitumen deforms back into the grips. In the worst case, the membrane pulls free. With the extensometer there is no slippage. Thus the crosshead movement measurements are not as reliable as the extensometer measurements. However, for large strains the crosshead measurements are necessary to determine ultimate elongation when the membrane stretches past the limits of the extensometer. The decision to present load-strain vs. stress-strain data results from the variations exist in specimen thickness. These variations produce variations in reinforcing fabric cross-sectional ratios that influence test results. Hence, to provide more direct comparison between membranes, all tensile strengths are given in load per unit width of specimen.
## APPENDIX D: PUNCTURE TEST RESULTS

**Penetration force (lb)**

<table>
<thead>
<tr>
<th>Membrane manufacturer</th>
<th>Test number</th>
<th>Average</th>
<th>Standard deviation</th>
<th>Coefficient of variation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Test set 1</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Soprema</td>
<td>160 124 168 180 166 152 158 154 148 172 168 172 156 168 157 164 130 152 157 148</td>
<td>157.7</td>
<td>13.6</td>
<td>8.6</td>
</tr>
<tr>
<td>NEI</td>
<td>84 96 70 104 82 92 88 79 100 84 86 87 95 80 80 90 90 96 98</td>
<td>88.4</td>
<td>9.3</td>
<td>10.5</td>
</tr>
<tr>
<td>Polyguard</td>
<td>90 72 76 80 66 80 98 62 66 62 64 58 68 68 82 63 81 63 64 68</td>
<td>71.6</td>
<td>10.6</td>
<td>14.8</td>
</tr>
<tr>
<td>Royston</td>
<td>50 46 48 52 48 48 50 44 46 46 56 54 56 50 54 56 46 52 48 56</td>
<td>50.3</td>
<td>4.0</td>
<td>7.9</td>
</tr>
<tr>
<td>Protecto Wrap</td>
<td>44 40 34 40 44 38 36 42 44 34 30 40 36 40 40 41 32 42 38</td>
<td>38.5</td>
<td>4.1</td>
<td>10.7</td>
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<tr>
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<td>36.5</td>
<td>6.7</td>
<td>18.3</td>
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</tr>
<tr>
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<td>40 34 38 40 42 44 39 38 36 37 40 37 43 46 42 36 37 44 44 36</td>
<td>39.7</td>
<td>3.4</td>
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</tr>
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<td>36.0</td>
<td>6.3</td>
<td>17.5</td>
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<td><strong>Test set 3</strong></td>
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<td>37.5</td>
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<td>19.0</td>
</tr>
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<td>W.R. Grace</td>
<td>40 24 36 22 56 50 52 40 40 36 34 40 42 46 38 36 40 34 38 36</td>
<td>39.0</td>
<td>8.1</td>
<td>20.7</td>
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APPENDIX E. MODIFIED-CUP TEST RESULTS

Test Conditions: 84°F for an average vapor pressure difference of 1.1316 in. of Hg. The sample area in all cases was 0.0985 ft² and the length of the test was 560 hours.
APPENDIX F. WET-CUP TEST RESULTS

Test Conditions: 84°F and 16% RH for an average vapor pressure difference of 0.9824 in. of Hg. The sample area in all cases was 0.0985 ft² and the length of the test was 720 hours.

a. NEI

b. Polyguard

c. Protecto Wrap

d. Royston

e. Soprema

f. WR Grace