

DOCUMENTATION AND TESTING OF NINETEENTH-CENTURY  
LIMEWASH RECIPES IN THE UNITED STATES

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## Abstract

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Limewash, a mixture of lime putty and water used as a surface coating, has served as an architectural finish throughout the world for thousands of years, but little has been written about the use of this material in the United States. Historic sources cite the use of limewash in a number of different environments, including farm buildings, military buildings, and residences. However, it is not known whether limewash was commonly used throughout the United States or only in certain regions. This thesis attempts to answer that question through historical research by examining published materials that specifically mention limewash.

The history of publishing and the current availability of sources were major factors that defined the parameters of this research. Over 1,000 sources mentioning limewash were documented in order to examine spatial and temporal trends in limewash use. Most of the historic publications describe (then) new recipes for limewash intended to improve its performance. These recipes were documented and analyzed according to the additives listed. This thesis examines the various recipes and the intended effects of a number of additives.

Testing was performed to understand basic properties of limewash and additives and to ascertain the legitimacy of historic recipe claims. The tests utilized ASTM methods for adhesion, chalking resistance, and water-vapor transmission. The technical research, in combination with collection of historic paint recipes, seeks to answer questions about the historic use of limewash. Based on these findings, this thesis suggests appropriate uses for limewashes or historic limewash recipes based on testing rather than popular beliefs. This will allow preservationists and practitioners alike to be more informed when choosing a limewash for historic structures.

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## 1. INTRODUCTION

Limewash has reemerged as a viable paint option in recent years. This architectural finish is seen as appropriate to certain historic periods, and perhaps more importantly, compatible with certain historic building materials. Limewash is touted as a useful coating for historic fabric, because it is breathable, sacrificial, and antiseptic. Despite these benefits, there is not a large amount of research on limewash in the United States. The history of the material is not well known, and little quantitative information on the properties of limewash exists. This thesis seeks to expand the literature on limewash use in the United States and broaden the understanding of how it may be used on historic structures today.

The initial idea for this thesis came from the author's summer 2014 internship with the National Park Service (NPS) at Governors Island National Monument. During this internship, historic limewash and its use in military fortifications were researched with a focus on Castle Williams, a fort on Governors Island. Part of the interest to continue studying limewash stemmed from the limited amount of published information on the history of its use found during the internship. Therefore, one preliminary goal of this thesis was to add to the literature on this subject.

In its most basic form, limewash is a mixture of lime putty and water, used as a paint. Limewash has been frequently used to coat masonry, plaster, and wood. It cannot easily be applied to non-porous materials such as metal or glass, because limewash typically needs a porous substrate on which to adhere. Historically, limewashes were used for practical purposes such as protecting a building material or providing sanitary surfaces. This meant that limewashes were most often used for basements, storage houses, fences, and barns. Many historic sources discuss agricultural applications of limewash (e.g., for fences and barns), but residential

buildings were often limewashed as well. As both interior and exterior surfaces could be limewashed, it was certainly one of the cheaper paints available, leading to its prevalence in America.

While this thesis does not examine limewash use in other parts of the world, it is important to note that its use has been widespread and extends back a long time. Some sources state that its use goes all the way back to ancient Egypt in the temples of the Pharaohs.<sup>1</sup> There is much more published information in English regarding historic limewash in European countries. For example, the white facade of Italian 11th century Romanesque churches were said to be whitewashed,<sup>2</sup> and there is a story of how King John of England ordered all the houses on the Thames to be limewashed inside and out after a fire in 1212.<sup>3</sup> These of course are limited vignettes of limewash use worldwide. It has also been used in many other places with diverse climates ranging from Yemen<sup>4</sup> to Kazakhstan<sup>5</sup> to Brazil<sup>6</sup> to Portugal<sup>7</sup> plus many more. There has never been an aggregation of data from different countries or continents regarding limewash, but this would certainly make for a useful resource to better understand the broader history of

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<sup>1</sup> Klemenc, Stacey Enesey. 2003. 'Decorative Paint, Lime wash', *Paint PRO: The Professional Paint & Decorating Contractor's Journal*, 5(1): accessed online January 15, 2015. <[http://www.paintpro.net/Articles/PP501/PP501\\_Limewash.cfm](http://www.paintpro.net/Articles/PP501/PP501_Limewash.cfm)>.

<sup>2</sup> Barry, Fabio. 2011. 'Painting in Stone: The Symbolism of Colored Marbles in the Visual Arts and Literature from Antiquity until the Enlightenment'. Ph.D. Dissertation. Columbia University, 346.

<sup>3</sup> Davies, Gill. 2006. *Five Hundred Buildings of London*. New York: Black Dog & Leventhal Publishers, Inc., 566.

<sup>4</sup> Jerome, Pamela, Chiari, Giacomo, and Borelli, Caterina. 1999. *APT Bulletin*. XXX(2-3):39-48.

<sup>5</sup> As a protective layer for the loess steppe area of Shaul Der.

Fodde, Enrico. 2009. 'Traditional Earthen Building Techniques in Central Asia', *International Journal of Architectural Heritage*, 3(2): 145-168.

<sup>6</sup> In Costa da Lagoa, Santa Catarina.

Kanan, I. 2013. 'Researching the traditional building culture of Costa da Logoa, Santa Catarina, Brazil'. In *Vernacular Heritage and Earthen Architecture: Contributions for Sustainable Development*, Correia, M., Carlos, G. and Rocha, S. (eds.) 77-82. London: Taylor & Francis Group.

<sup>7</sup> In Carrasqueira, a fishing hut village.

Jorge, F. and Bruno, P. 2013. 'Carrasqueira - fishing harbour on stilts, hut village in Portugal'. In *Vernacular Heritage and Earthen Architecture: Contributions for Sustainable Development*, Correia, M., Carlos, G. and Rocha, S. (eds.) 77-82. London: Taylor & Francis Group.

limewash. Due to resource and time limitations however, research for this thesis solely focused on limewash in America.

The prevalence of limewash is partially due to its beneficial properties (breathable, sacrificial, antiseptic), but the low cost, ease of use, and prevalence of limestone worldwide also contributed to the frequency of its use. These qualities are precisely why it is difficult to learn about the history of limewash, as it was an inexpensive, everyday surface coating that would eventually wear away. Thus, there is much less documentation of its use than other more expensive finishes, such as oil paint, and less physical evidence of limewash remaining on historic buildings. For this thesis, research into published historical references to limewash was crucial to gain an understanding of its use and place in American architectural history.

The initial intention of this thesis was to examine the use of limewash in the United States as a whole. However, research into early American usage in the 17th and 18th centuries proved difficult as there were few detailed sources. Many more sources specified how, when, where, and why it was used from the 19th century onward. Therefore, research was focused on these 19th century published resources. To begin research, information was gathered by searching through thousands of published documents that mention the use of “limewash” or “whitewash.”<sup>8</sup> The sources included journals, homeowners and builders’ guides, household recipe (or receipt) books, newspapers, and personal correspondence. Most of the documents were found in online databases including Google Books, Cornell University Library’s Making of America Collection, and the Library of Congress’ Chronicling America database. From those sources, patterns were analyzed to help determine how people used limewash. Many of the references were presented in conjunction with a recipe intended to alter its properties. Since limewash was not a durable material, recipes were developed to improve its qualities, for

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<sup>8</sup> The terms “limewash” and “whitewash” are discussed in detail in Section 2.1.



example making it waterproof, more durable, or its substrate incombustible. These recipes contained additives such as salt, milk, sugar, linseed oil, whiting, zinc sulfate, alum, flour, and even blood. Occasionally, recipes enumerated the pigments that could be added to limewash, but this thesis does not delve into pigment research or testing. During research, each recipe was recorded in a spreadsheet broken down by ingredients, year, and location.

A second large spreadsheet for each mention of limewash in conjunction with a location was created to aid with analysis. The two spreadsheets allowed for the examination of trends in limewash use. From that information, maps were made using ArcGIS to show where and when limewash use was mentioned in the 19th and early 20th centuries. These maps are discussed in Section 3.2. In addition to this quantitative data, any useful qualitative information that came from looking through the thousands of historic sources was noted. Much of the research focus was on the performance of pure limewash versus modified limewash, as well as what functions additives were thought to have.

In addition to the previous lack of historical research into limewash, there is not much testing data available. Because of this, a technical component was added to the thesis. One study available was the National Center for Preservation Technology and Training's 2003-2005 project where they tested the durability of three limewash recipes prepared with different types of lime (dry hydrate vs. lime putty, high calcium vs. dolomitic).<sup>9</sup> NCPTT's testing became a model that was adapted while the preliminary testing plan for this thesis was formulated. Of course, a great deal of historic research was necessary to fill in the specifics of the testing.

The information gathered from research was used to inform the testing plan, which was created to evaluate the function of different limewash additives. Salt, sugar, milk, zinc sulfate,

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<sup>9</sup> Jackson, Sarah. 2005. 'Study on the Durability of Traditional and Modified Limewash Recipes'. Natchitoches, LA: U.S. Department of Interior National Park Service, National Center for Preservation Technology and Training.

alum, whiting, and ground rice were the additives tested. Historic recipes made claims about the function of these additives, which are detailed in Chapter 4. For example, one claim was a limewash with sugar added “makes it adhere splendidly... drops that fall to the floor or window will not wash off.”<sup>10</sup> These claims informed the testing plan design for this thesis; American Society for Testing and Materials (now ASTM International) standard test methods were chosen to assess the validity of historic claims. Three test methods--adhesion, chalking resistance, and water-vapor transmission--were performed on one pure limewash and five limewashes with additives based on historic recipes. The full testing plan is detailed in Chapter 5, and the procedure is described in Chapter 6. The plan details why the specific recipes, tests, number of coats, and materials were chosen. Those choices were made based on historic research and the many sources referencing the use of limewash. Each step of the testing process was photographed and the results recorded.

The tests provided some conclusions about the performance of historic limewash recipes, proving some historic claims true and others questionable. Chapter 7 contains the full test result analysis, while Chapter 8 compares the results for each recipe to the historic claims. Chapter 8 also posits how this research may be taken further and used by preservationists, conservators, and practitioners. There are still many unexplored areas of limewash research that could lead to valuable new information on the subject. As a contribution to that research, this thesis seeks to expand the literature on the use of limewash in the United States in the 19th and early 20<sup>th</sup> centuries, while putting those 19th-century notions of limewash to the test.

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<sup>10</sup> ‘Sugar and Mortar’. *The Manufacturer and Builder*, 22(3): 57, 1890.

## 2. LIMEWASH BASICS

In order to understand the way in which limewash functions, it is important to understand what a limewash is at its most basic. Its properties and composition historically made it an important coating for a number of practical reasons. Lime is what gives limewash its properties. Lime is used as a building material in many forms, and limewash is one of the simplest.

### 2.1 Limewash terminology

The terms “limewash” and “whitewash” are often used interchangeably to refer to the mixture of lime and water discussed in the previous section. Today, most people in the conservation and preservation field use the term “limewash.” Historically, “whitewash” was by far the most common term. Whitewash can be a confusing label in historical sources, because it could be used to refer to a pure limewash, a limewash modified with additives, a paint made with whiting (crushed calcium carbonate), or sometimes even other types of white paint. The vast majority of the recipes collected for this thesis refer to whitewash. However, limewash will be the primary term used to describe the mixture, since that is the more common term used today.

### 2.2 What is a limewash?

In its most basic form, limewash is a mixture of lime putty and water, used as a surface coating. Lime putty is calcium hydroxide, which is made by adding water to quicklime (calcium oxide). Quicklime is produced by burning a source of lime.<sup>11</sup> Historically, limewash was made by adding water to quicklime, straining the mixture through a sieve, then letting the resulting putty age (these two steps would make a mixture that was easier to work and potentially more durable but were not always done), and finally adding more water to thin the mixture to a whole

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<sup>11</sup> The most common source of lime is limestone, but other sources such as oyster shells can also be burned to produce quicklime.

milk or cream-like consistency.<sup>12,13</sup> Historic sources do not give proportions for lime putty and water in order to achieve that consistency. It must be assumed that limewash was so common in the 19th century that people did not need instructions on how to prepare the basic material.

Today, limewash can also be made with dry hydrated lime, a product whose use began after the turn of the 20th century.<sup>14</sup> Hydrated lime is created by manufacturers through crushing quicklime, adding a controlled amount of water to form the hydroxide(s), which are classified to meet specifications.<sup>15</sup> It is often considered easier to use than lime putty, because it comes in the form of a powder. Hydrated lime can be mixed with water to create a limewash, but some modern sources claim that when it is created this way, limewash does not perform as well as one mixed with lime putty that has been fully slaked.<sup>16</sup> In their study, NCPTT found that there was little difference in performance of hydrated lime and lime putty, with lime putty having only a slight edge.<sup>17</sup> However, since hydrated lime was not used historically, the decision was made to prepare all limewashes for this thesis with lime putty. Graymont Niagara Lime Putty, the lime putty used by NCPTT in their study, was chosen specifically because it had tested well for them and so that the results could be compared.

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<sup>12</sup> Walker, P.H. 1913. 'The Use of Paint on the Farm'. In *Eighteenth Biennial Report*, 142-156. Topeka: Kansas Department of Agriculture.

<sup>13</sup> Storke, E.G. 1859. *The Family and Householder's Guide*, Auburn, NY: The Auburn Publishing Company.

<sup>14</sup> 'History of Lime in Mortar', Graymont Limited website, accessed March 22, 2015, <<http://www.graymont.com/en/markets/construction/building/mortar-applications/history-lime-mortar>>.

<sup>15</sup> 'How Lime is Made', The National Lime Association website, accessed March 18, 2015, <<http://lime.org/lime-basics/how-lime-is-made/>>.

<sup>16</sup> 'Lime Binders', US Heritage Group website, accessed April 3, 2015, <<http://usheritage.com/binders/>>.

<sup>17</sup> Jackson, Sarah Marie. 2007. 'Durability of Traditional and Modified Limewashes', *APT Bulletin: Journal of Preservation Technology*, **38**:2-3, 19-28.

### 2.3 Application and curing

The creation and curing of limewash is completely dependent on the nature of lime itself. Much has been written about the lime cycle, so it will not be discussed in great detail here.<sup>18</sup> The basic principle is that calcite limestone ( $\text{CaCO}_3$ ) undergoes a compositional cycle through calcining (heating), slaking (adding water), and carbonation to finally return to its original chemical form. The process of applying a limewash begins after the slaking step in the lime cycle, adding more water to the fresh putty to create the proper consistency before applying it to the substrate. Carbonation, the final step in the lime cycle, is the means by which the mixture cures. Limewash takes in carbon dioxide from the air in order to solidify on the substrate.

When a dolomite limestone is used, the cycle is similar but deviates somewhat, especially at the carbonation phase. Dolomite is  $\text{CaMg}(\text{CO}_3)_2$  and goes through the same initial steps as calcite:  $\text{CaMg}(\text{CO}_3)_2 > \text{CaO}, \text{MgO} > \text{Ca}(\text{OH})_2, \text{Mg}(\text{OH})_2$ . Dolomitic lime differs in that the cured products can consist of  $\text{CaCO}_3$ ;  $\text{Mg}(\text{OH})_2$ ;  $\text{MgCO}_3$ ,  $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ ;

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<sup>18</sup> From the British Lime Association website <[http://www.britishlime.org/education/lime\\_cycle.php#level01](http://www.britishlime.org/education/lime_cycle.php#level01)>, this is a full and detailed description of the chemical process of lime cycle:

“Burning limestone/chalk occurs at roughly 900°C. Kilns are often heated to 1200°C in order to ensure the core regions of all the rock pieces also reach this temperature. The reaction for the thermal decomposition of calcium carbonate is as follows:  $\text{CaCO}_3 + \text{heat} \rightarrow \text{CaO} + \text{CO}_2$  This chemical reaction produces quicklime. Different kilns are used for different types of limestone/chalk and for different types of product. Quicklime's principal component is calcium oxide. Its quality often depends on a number of certain factors including physical properties, reactivity to water and chemical composition. As the most readily available and inexpensive alkali, quicklime plays an essential part in a wide range of industrial processes. Adding water to quicklime produces an exothermic reaction (gives out heat) and hydrated lime. The reaction for the hydration of quicklime is as follows:  $\text{CaO} + \text{H}_2\text{O} = \text{Ca}(\text{OH})_2 + \text{heat}$  Hydrating plants are relatively complex and can be fed with surplus grades of quicklime. Hydrated lime, despite its name is essentially dry and generally contains less than 1% of unreacted water. An estimated 10% to 15% of quicklime produced in developed countries is converted into hydrated lime (a percentage which is probably larger for countries which do not have a large steel industry). Slaking lime involves the production of a dispersion of calcium hydroxide in water, creating a product known as milk of lime or lime putty. The recarbonation process is essentially the opposite of the calcining/burning process. Both quicklime and hydrated lime, when exposed to the air for long periods, begin to draw in carbon dioxide from the atmosphere. This therefore replaces the oxide component of the chemical and turns the lime roughly back to its original state - being limestone or chalk. The formula is as follows:  $\text{Ca}(\text{OH})_2 + \text{CO}_2 = \text{CaCO}_3 + \text{H}_2\text{O}$  Recarbonation can occur in a number of lime based products and in many cases acts as a benefit. For example, when the process occurs in lime mortar it effectively increases the strength of the final bond and acts as a self healing solution if any cracking occurs.”

$\text{Mg}_2(\text{CO}_3)(\text{OH})_2 \cdot 3\text{H}_2\text{O}$ ;  $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 5\text{H}_2\text{O}$ ; and  $\text{Mg}(\text{HCO}_3)(\text{OH}) \cdot 2\text{H}_2\text{O}$ .<sup>19</sup> Much of the lime produced in the United States is actually dolomitic.<sup>20</sup> For this reason, and because NCPTT used dolomitic lime putty for their study, dolomitic lime was chosen for the testing in this thesis.

## 2.4 Properties

The advantageous properties of a limewash are the primary reasons it was used so frequently in the 19th century (and before). One major benefit is the antiseptic nature of limewash. The alkalinity of the coating prevents bacterial growth, which made it a popular choice for farms, outhouses, basements, and areas that could harbor germs, mold, and bacteria.<sup>21,22</sup> However, this benefit would not last indefinitely; limewash must be renewed to ensure its effectiveness as an antiseptic.<sup>23</sup> In 1897, J.W. Rigterink wrote his thesis for Michigan State Agricultural College on limewash's effect on bacteria.<sup>24</sup> Rigterink concluded that limewash prevented the growth of four of the five bacterial strains he tested. This study from 1897 is one of the few laboratory studies that exist on this property of limewash; most of what is known comes from tradition, unsubstantiated writings, and anecdotes.

In this vein, a meeting of the American Veterinary Medical Association in 1899 discussed the antiseptic benefits of limewash in barns and stables. One doctor stated that he knew a group of horses were infected with “lung plague” but once the stables were limewashed, they never had to be disinfected again.<sup>25</sup> He went on to say that limewash was much more cost

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<sup>19</sup> Hartshorn, Heather. 2012. ‘Dolomitic Lime Mortars: Carbonation Complications and Susceptibility to Acidic Sulfates’. Masters Thesis. Department of Historic Preservation, Columbia University.

<sup>20</sup> Ibid., 2.

<sup>21</sup> Walker, 142-156.

<sup>22</sup> ‘Preparation of Whitewash’, *The Louisiana Democrat*, August 14, 1884. Chronicling America Historic American Newspapers database, Library of Congress.

<sup>23</sup> ‘Whitewashing - A Whitewash’, *New England Farmer*, XII(6): 268, 1860.

<sup>24</sup> Rigterink, J.W. 1897. ‘The Action of Whitewash Upon Bacteria’. Thesis. Michigan State Agricultural College.

<sup>25</sup> Grange, E.A.A. 1899. *Proceedings of the American Veterinary Medical Association*, Vol. 36. Ithaca, NY: American Veterinary Medical Association.

effective and less harmful for the animals than other alternatives. There are many similar stories of farmers limewashing their poultry houses and other storage facilities to promote healthfulness. Some farmers' bulletins even suggest limewashing the poultry houses twice a year.<sup>26,27</sup> This practice was said to prevent lice and other insects from infecting the chickens and "more than pay for the trouble and expense incurred."<sup>28</sup> People truly believed in its sanitary benefits, associating its appearance with "cleanliness and neatness."<sup>29</sup> The belief in an ability to deter illness and disease is repeated in many journals, books, and bulletins and was presumably a major factor in limewash's popularity.

Another benefit is that limewash acts as a sacrificial coating for its substrate, protecting the material underneath. Some modern sources even claim that it can help to consolidate deteriorating stone.<sup>30</sup> Its breathability is one of the reasons that limewash is a good sacrificial coating. Limewash allows water to transmit through the layer without getting trapped behind it in the substrate. By contrast, some other paints can prevent the transmission of water and instead cause water to be retained in the substrate, which leads to deterioration. Conservators often tout the breathability of limewash as one of its main benefits. While this is true when used without additives, one of the hypotheses of this thesis is that additives (and the multiplicity of coats) might negatively affect the breathability. Water-vapor transmission dishes were assembled to test this hypothesis. The test procedure and results are detailed in Chapters 6 and 7, respectively.

While it protects the substrate from damage, limewash wears away over time. Due to its transience, limewash must be renewed more frequently than many other paints. Often sources

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<sup>26</sup> Lloyd, W.B. 1894. *Lloyd's Modern Poultry Book*. Chicago: Howard & Wilson Publishing, Co., 113.

<sup>27</sup> Stewart, Dan. H. 1892. 'Lime', *Western Garden and Poultry Journal*, 4(5): 128.

<sup>28</sup> 'Lime and Whitewash', *Rocky Mountain Husbandman*, February 16, 1882, 1. Chronicling America Historic American Newspapers database, Library of Congress.

<sup>29</sup> *Ibid*, 1.

<sup>30</sup> Mold, P. and Godbey, R. 2005. 'Limewash: Compatible Coverings for Masonry and Stucco'. *International Building Lime Symposium*, National Lime Association, Orlando, Florida, 9-11 March 2005.

suggested repainting once a year, and some households even considered it a yearly ritual.<sup>31</sup> This extra time and effort needed to maintain limewash may be considered a disadvantage compared to other more long lasting paints. However, repainting allows for the renewal of limewash's sanitary benefits as well. In order to help prolong service life, many modified recipes were developed. These recipes and additives are discussed in Chapter 4.

Finally, some of the biggest historical advantages of limewash were actually extrinsic properties; it was inexpensive and easy to make. A bushel of lime (about 70-80 pounds depending on the region) cost only \$0.30 in 1882, so it was affordable for the home user.<sup>32</sup> Lime was commonly used on farms in a number of capacities and readily available to make limewash. Its ease of mixing and use meant that it was made by farm workers and homeowners rather than professional painters. Historically, it was a vernacular substance, and as such, the knowledge of how to make a limewash was well known in the United States. This is evidenced by the fact that articles and recipes from the 19th century never give proportions for mixing a pure limewash as mentioned earlier.

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<sup>31</sup> Moss, Roger W. 1994. *Paint in America: The Colors of Historic Buildings*, Washington D.C.: Preservation Press, National Trust for Historic Preservation, 15.

<sup>32</sup> Hodgson, Frederick Thomas. 1882. *The Builder's Guide and Estimator's Price Book*. New York: The Industrial Publication Company, 29.



### 3. LIMEWASH HISTORY IN AMERICA

#### 3.1 Early history

Limewash was used in the United Kingdom for at least a few hundred years before colonists came to America.<sup>33</sup> Therefore, this knowledge would likely have traveled with them. This idea is supported by paint analysis on surviving early American buildings in Colonial Williamsburg. Unfortunately, there are not many thorough paint analysis studies publically available, but some do exist. Studies provided by Colonial Williamsburg indicate that limewash was used on buildings in addition to oil paints.<sup>34</sup> A large proportion of the analyzed limewashes were shown to contain protein and carbohydrate additives. Even though historic recipes from this time are rare,<sup>35</sup> these analyses show that additives were used even then. It should be noted that most of the surviving buildings are examples of wealthier residences and high-end businesses, which were likely to have used oil paints. Structures such as fences, outbuildings, and simpler homes were the types of buildings likely to utilize limewash, but fewer of these survive today. The fact that many affluent residences used limewash in some situations is a strong indication that the more numerous modest structures likely would have as well.

Little writing about limewash from the early American period exists (or at least is accessible). A few sources are known and give some understanding of limewash use in the 16th and 17th centuries. For example, the National Park Service states in *Preservation Brief* 28 that

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<sup>33</sup> Davies, 566.

<sup>34</sup> These reports can be found in the Colonial Williamsburg Digital Library. <<http://research.history.org/DigitalLibrary/>>. Two such reports that mention limewash layers are cited below:

- Loeblich, Natasha K. 2007. 'Cross-Section Microscopy Analysis of Exterior Paints: Palmer House, Williamsburg, Virginia'. Colonial Williamsburg Foundation Library Research Report Series - 1737, Colonial Williamsburg Foundation Library.
- Travers, Kirsten E. 2012. 'Cross-Section Microscopy Analysis of Interior Paints: Margaret Hunter Shop, Williamsburg, Virginia'. Colonial Williamsburg Foundation Library Research Report Series - 1747, Colonial Williamsburg Foundation Library.

<sup>35</sup> No recipes from that time were found during research for this thesis. However, that does not rule out the possibility that some written recipes do exist.

by the mid-17th century, limewash was commonly applied to interior walls.<sup>36</sup> Additionally, Roger W. Moss states in *Paint in America: The Colors of Historic Buildings* that in 1785, Francis Hopkinson wrote a letter detailing the yearly process of limewashing the walls in his home.<sup>37</sup> Even though the written evidence is thin, this draws a line from the 17th-century material evidence through the late 18th century. It is easier to pick up the story of limewash in the 19th century, when written documentation was much more common.

### 3.2 19th-century limewash

By the start of the 19th century, some sources, such as newspapers in England, were publishing recipes for limewash. In April and July of 1803, the *Morning Chronicle* in London published two recipes for modified limewashes, one containing sea salt and the other containing milk, linseed oil, whiting, and rosin.<sup>38</sup> The latter recipe was touted as “A New Method of Making Paint invented by Mr. Devaux, a member of the French National Institute.”<sup>39</sup> In the same year, an American newspaper in New York, *Commercial Advertiser*, published the same recipe for the paint by Mr. Devaux.<sup>40</sup> Duplication of these recipes indicates that ideas and innovations for limewashes were shared, copied, and disseminated between England and the United States, as well as other locations such as the French National Institute. Prior to the late 18th and early 19th centuries though, limewash recipes were not often written down or published. Ingredients were predominantly handed down by oral or local tradition.

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<sup>36</sup> Chase, Sara B. 1992. ‘Painting Historic Interiors’. *Preservation Brief* 28. U.S. Washington, D.C.: Department of the Interior, National Park Service.

<sup>37</sup> Moss, 14.

<sup>38</sup> *Morning Chronicle*, April 16, 1803, in Rita Susswein Gottesman, *The Arts and Crafts in New York, 1800-1804*, The New-York Historical Society, 1965.

<sup>39</sup> *Morning Chronicle*, July 16, 1803 [also in *Commercial Advertiser*, June 29, 1803], in Gottesman, Rita Susswein, *The Arts and Crafts in New York, 1800-1804*, The New-York Historical Society, 1965.

<sup>40</sup> Ibid.

As the 19th century went on, more and more sources published information about limewash. This is neither altogether unsurprising, nor unique to limewash. The growth of the print industry in America and increase in transportation meant that more journals and books were published in general.<sup>41</sup> Books and journals on building, including paint and other finishes, increased significantly in number. Some of these publications contained information on limewash. Some recipes were published from 1800 to 1840, but the predominant period for limewash recipe publication was 1840 to 1900.

The increase in written limewash references allows for analysis of how, when, and where it was used in the United States. Of course, there are limitations when performing an analysis based on only published references. Published sources are not necessarily representative of limewash use in all of America, but it is at least an indication of basic trends.

### *3.2.1 Where was limewash used?*

The following map shows limewash use references by location. To create this map, over 1,000 books, journals, and newspapers that mentioned the use of limewash were identified.

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<sup>41</sup> Hutchinson, Peter. 2008. 'Magazine Growth in the Nineteenth Century'. *A Publisher's History of American Magazines*. The Magazinst website, accessed April 6, 2015, <[http://www.themagazinst.com/Magazine\\_History.html](http://www.themagazinst.com/Magazine_History.html)>.

## Limewash References by Location

19th century - early 20th century

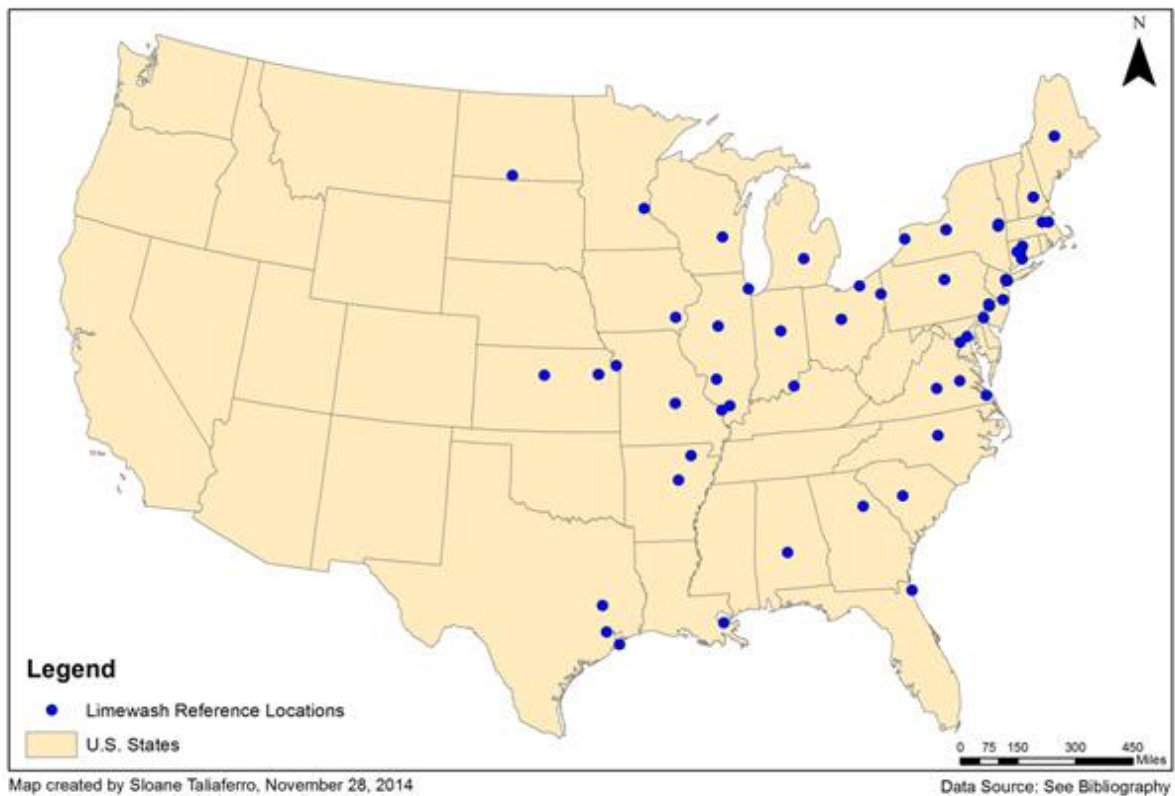


Figure 1. Limewash references by location

From these sources, each location that was associated with one or more references was then mapped.<sup>42</sup> The map shows a scattered used of limewash in the eastern half of the United States with a more concentrated proportion in the northeast. This seems to indicate that limewash was not used in the western half of the country. However, there are most likely mitigating factors involved that would call this into question, as limewash was definitely used on adobe buildings in the West and Southwest.<sup>43,44,45</sup> No references to limewash in those areas were collected,

<sup>42</sup> This could be a town, county, or state. If the location mentioned was a county or state, I let the map randomly choose where to place the dot within the specified county or state.

<sup>43</sup> La Motte, A.V. 1897. *Overland Monthly and Out West Magazine*, XXX(177): 239.

<sup>44</sup> Clifton, J.R. 1977. *Preservation of historic adobe structures: a status report*, Washington, D.C.: Department of Commerce, National Bureau of Commerce.

<sup>45</sup> Deverell, W. 2004. *Whitewashed Adobe*, Los Angeles: University of California Press.

because few sources mention the use of limewash in the Western states and territories due to a number of factors. To begin with, many of the references in the East came from people writing to journals with questions about limewash, but the circulation of these journals may not have reached the West. Additionally, some territories in the western half of the United States were not even admitted into the Union until the late 19th century or early 20<sup>th</sup> century. This could have had an effect on their likelihood to read and to write to publications, even if they were using limewash. Therefore, this map cannot perfectly represent America's use of limewash in the 19th and early 20<sup>th</sup> centuries, but instead it represents a snapshot of where people who communicated and learned new ideas about limewash lived.

In order to understand the density of limewash references in the Northeastern states, the following map shows the same information but in the form of a dot density map by state.

## Density Map of U.S. Limewash Use References 19th century - early 20th century

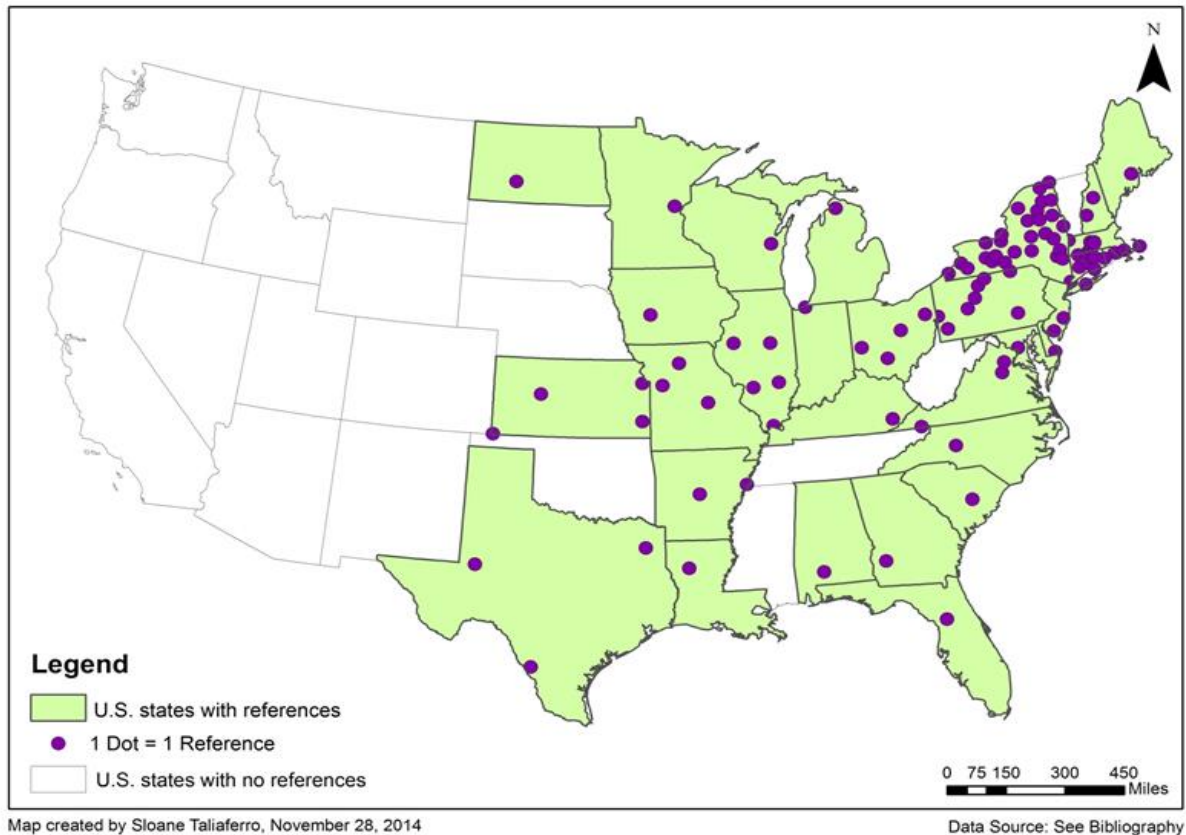


Figure 2. Density map of U.S. limewash use references

The map shows a large proportion of people in the Northeast were writing about limewash use. This is most likely due to the greater amount of people living in that area, as well the fact that the publishing industry was focused there. Nonetheless, these maps yield quantitative information about where 19<sup>th</sup>- to early 20<sup>th</sup>-century people were communicating about limewash.

### 3.2.2 *When was limewash used?*

These same data points can be used to look at temporal trends in the use of limewash. When analyzing these maps, the same limitations discussed in Section 3.2.1 apply. The following eight maps break down the previously discussed limewash references by decade.

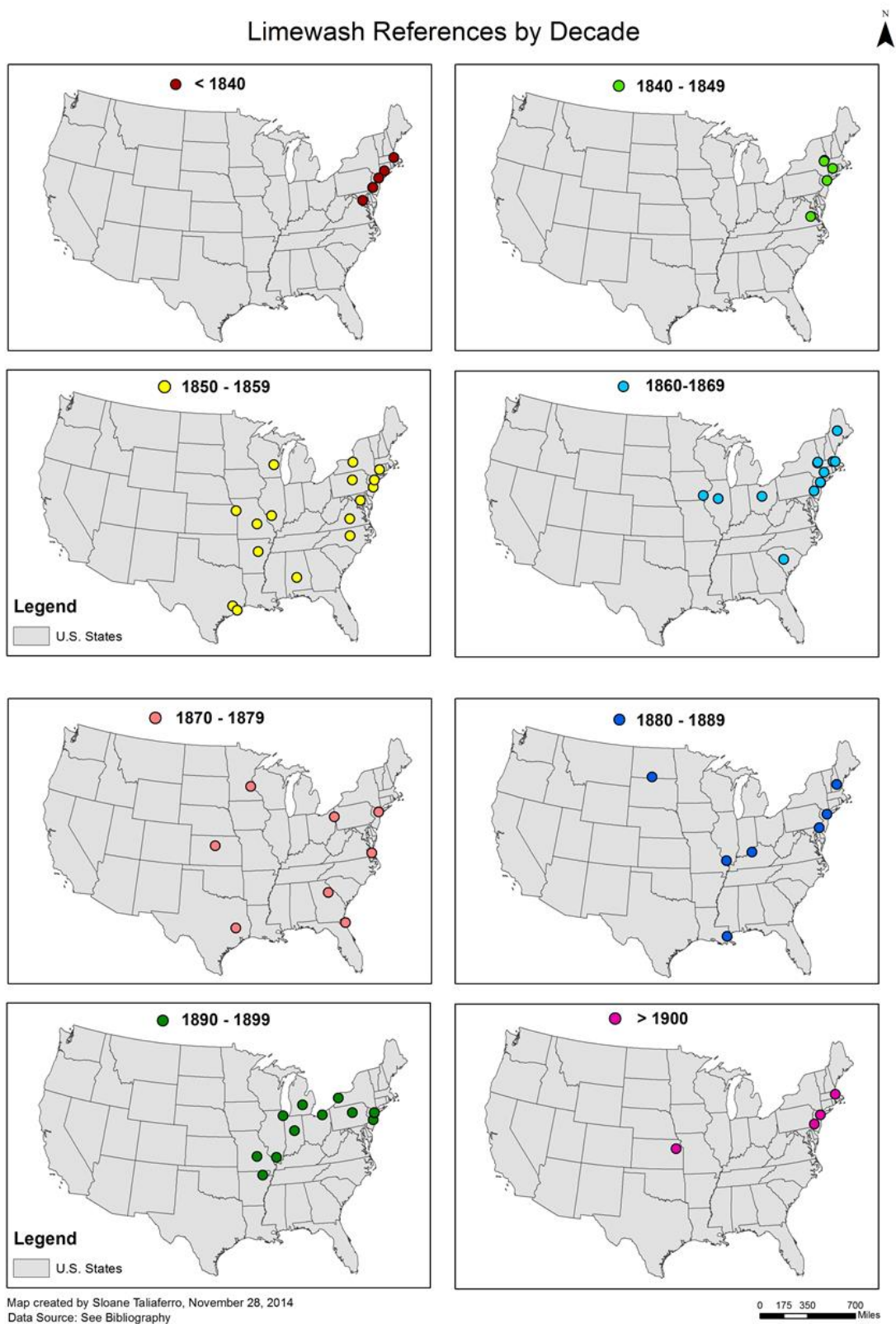


Figure 3. Limewash references by decade

The maps show that published references to limewash use increased in number and reach in the second half of the 19th century. In the first half, there were far fewer published references, and the ones that did exist were concentrated in the Northeast. The 1850s saw an explosion of published references and the largest amount of any decade in the 19th century. Until 1900, people from the Eastern half of the country continued to write to journals and newspapers. However, after 1900, there was a decline in publications writing about limewash, which corresponded to an overall decline of limewash in the 20<sup>th</sup> century. This decline will be discussed in Section 3.3.

### 3.2.3 How was limewash used?

The same sources used in the previous maps also yield some important information about *how* people were using limewash. Most references from the maps were, in some way, related to recipes. A significant number of people would write to journals such as *Scientific American* and *Manufacturer and Builder* asking for limewash recipes. Sometimes, people stated that they wanted a limewash recipe for a specific purpose such as one appropriate for wood or for waterproofing, but others were simply asking for a “good” or “durable” recipe. One correspondent who wrote to *Manufacturer and Builder* from Mayport, FL stated that he was looking for a “good, durable wash” for outdoor walls and fences that would withstand storms. The correspondent goes on to say that he had tried many recipes in the past that were presumably not satisfactory. One of the most interesting things about his letter was the opening statement, “You are probably aware how largely whitewash is used all throughout the South.”<sup>46</sup> While this anecdote from 1873 does not give any specific information about how many people in the South used limewash, this correspondent’s perception of whitewash was that it was common.

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<sup>46</sup> ‘Waterproof Whitewash’, *The Manufacturer and Builder*, 5(3): 71, 1873.



Additionally, his letter confirms that at least some people actually use the limewash recipes published in journals.

One of the most entertaining anecdotes about limewash came from a farmer in Highland, Illinois in 1856, who wrote to *Scientific American*. The correspondent complained that he tried a recipe for limewash that the journal had published in a recent issue, but he was upset that it only lasted half a day. The problem was that this recipe containing lime, water, and salt was licked off by his cattle, “as clean as if whitewash had never been put on!”<sup>47</sup> *Scientific American* goes on to say:

Well, we did not give the whitewash recipe for the purpose of salting his cattle; the salt was recommended to be mixed with the lime for the purpose of keeping the latter from scaling off by dry winds. The recipe, however, must be a most excellent one, when the very cattle of the prairies are led by instinct to *swallow* it.<sup>48</sup>

This amusing story helps to give more insight into how people used limewash in the 19th century. It demonstrates that people were actually using the recipes in published sources, sometimes apparently by trial and error, to determine what would work. This echoes the Mayport, FL man’s request for new recipes. Additionally, the cattle story demonstrates that limewash was a simple material, mixed and applied by the owner. It confirms the notion that limewash was a vernacular surface coating, used by everyday people on everyday structures.

### 3.3 Limewash decline in the 20<sup>th</sup> century

At the beginning of the 20<sup>th</sup> century, people were still writing about limewash. However, as time went on, most of the references and mentions became restricted to more rural communities. Agricultural bulletins and publications continued to write about uses for limewash

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<sup>47</sup> ‘Cattle and Whitewash’, *Scientific American*, **11**(39): 305, 1856.

<sup>48</sup> *Ibid.*, 305.

and the occasional recipe. Based on the agricultural bulletins of a number of U.S. states, it seems that farmers consistently used limewash throughout the first quarter and somewhat used it until the mid-20<sup>th</sup> century.

The National Lime Association published *Whitewash and Cold Water Paints* in 1939, a time when few individuals were writing about limewash. This pamphlet gives instruction for limewashing and ten recipes, some using the typical additives seen in the 19th century: salt, alum, molasses, whiting, linseed oil, and animal glue. However, *Whitewash and Cold Water Paints* also includes ingredients that do not seem to have appeared in any prior published recipes. These include trisodium phosphate, formaldehyde, carbolic acid, ammonia, borax, and casein (casein was present in older recipes but only in the unprocessed form of milk). Additionally, most recipes call for hydrated lime rather than slaked quicklime as in the 19th century. The National Lime Association does not explain what effects the ingredients such as trisodium phosphate or formaldehyde have on a limewash. They only state that “...new formulas were prepared and tested under actual weathering conditions. A number of these mixtures were found to be not only better, but easier to prepare and more economical than those which heretofore have been in common use.”<sup>49</sup> The assertion of ease is potentially misleading; while a limewash prepared with hydrated lime is certainly easier to prepare than slaking quicklime, the other “new” additives were ostensibly more difficult to obtain. By comparison, the 19th century recipes comprised common household (or farmhold) ingredients and could be easily mixed and applied by the owner. The use of these more modern additives may have been an attempt to compete with other types of newer, longer lasting paints. However, it is impossible to know whether or not people actually used the National Lime Association’s formulas.

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<sup>49</sup> National Lime Association. 1939. *Whitewash and Cold Water Paints*. Bulletin No. 304-D. Washington, D.C.: National Lime Association.

Regardless, new types of paint would certainly have been a major reason for the decline in limewash's popularity. Cement paint was one of limewash's direct competitors, beginning in the late 19th century and continuing more substantially into the 20<sup>th</sup> century. Like limewash, it was used as a low cost wall or fence coating and was good for fireproofing.<sup>50</sup> Unlike limewash, cement paint was cited as waterproof, long lasting, and strong without having to add other ingredients.<sup>51,52</sup> While cement paint was not the only surface coating to edge limewash out of regular use, its added benefits would suggest that people began to choose it over limewash.

In the 1940s, the introduction of emulsion paints using synthetic resin polymers represented a big change in the paint world, combining positive properties of both oil and traditional water-based paints.<sup>53</sup> As mentioned previously, limewashes (and some other water-based paints) are not durable or water resistant, but the emulsion paint was able to counteract this. The different types of emulsion paints available in the 20<sup>th</sup> century were styrene-butadiene, polystyrene, and acrylic paints.<sup>54</sup> By about 1960, they were widely manufactured and distributed, a major factor leading to the decline of limewash.<sup>55</sup>

To be clear, there were always many different types of paint to compete with limewash in the 19th century. Oil paints, kalsomine, distempers, and others had been alternatives to limewash depending on the function that the paint needed to serve. However, by the mid-20<sup>th</sup> century, it was not only the new paints with improved properties, but also new ways of obtaining paint that led to the decline of limewash. With the increases in technology and manufacturing, readymade paints became more easily available. Limewash had always been a coating mixed by the

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<sup>50</sup> 'Fire-Proof 'Paint', *The Manufacturer and Builder*, 7(4): 77-78, 1875.

<sup>51</sup> Moss, 235-236.

<sup>52</sup> 'Coating for Brick Walls', *The Manufacturer and Builder*, 2(11): 335-336, 1870.

<sup>53</sup> Standeven, Harriet A.L. 2011. *House Paints, 1900-1960: History and Use*. Los Angeles: The Getty Conservation Institute, 85.

<sup>54</sup> Ibid. 85-118

<sup>55</sup> Ibid., 117.

homeowner, but the new, relatively inexpensive emulsion paints boomed in the do-it-yourself market and became popular with professional painters and homeowners.<sup>56</sup> As limewash seems to have hardly been applied by professionals, the homeowner's shift to ready-made emulsion paints over limewash would have greatly reduced its use.

All of these factors led to a significant decrease in the use of limewash in the United States. Whereas it used to be so ubiquitous that how to mix a basic limewash was universally known,<sup>57</sup> it is only used for special projects in the U.S. today. Most of the limewash use now is for conservation projects in which limewash is believed to be the most compatible coating for a historic material or where it was the historic finish that preservationists deemed to be significant. The general population no longer has an understanding of limewash, its creation, use, or effects.

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<sup>56</sup> Ibid., 85.

<sup>57</sup> Most every published recipe only included special additives separate from lime and water. It was implied that everyone would already know how to mix a pure limewash.

## 4. RECIPES

As mentioned in the previous sections, published limewash recipes were common, especially in the 19th century. Adding different combinations of ingredients was intended to alter the basic properties of a limewash. Since it was not particularly durable and needed to be renewed frequently, many recipes were dedicated to changing that. Other recipes claimed that they would make the coating waterproof or render wood incombustible. Still, others merely stated that the recipe would create a “good” or “brilliant” limewash.<sup>58,59</sup> Interestingly, few recipes discuss enhancing or altering the antiseptic properties. This is presumably due to one of two reasons; either, users were satisfied with the inherent antiseptic characteristics or they did not consciously know which additives could increase antiseptic properties. A detailed list of the historical claims about different additives is discussed in Section 4.2. The following section discusses specific additives in recipes and where they were published.

### 4.1 Recipe sources and contents

Chapter 3 mentioned the lack of much solid information about early American limewash additives, but based on the paint analyses cited, it is clear additives were used. Unfortunately, there are not many written recipes from that period to study. Published recipes do not become common until the 19th century, especially in the second half. However, this is not surprising information since this coincides with the greater spread of information due to increases in printing technology and transportation happening on a larger scale during that time.<sup>60</sup>

Scientific journals, builders’ journals, homeowners’ and builders’ guides, family recipe books, newspapers, industrial encyclopedias, agricultural bulletins, and painters’ handbooks

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<sup>58</sup> ‘A Good Whitewash’, *Scientific American*, **13**(28): 224, 1858.

<sup>59</sup> ‘A Brilliant Whitewash’, *Southern Planter*, **3**(5): 104-105, 1843.

<sup>60</sup> Hutchinson, 2008.

were the types of sources to print limewash recipes. There does not appear to be any correlation between what types of recipes were published and their respective sources. However, it is certain that recipes were copied from one publication to another, as the exact same recipes appear in different sources over many years. For example, a recipe including lime, whiting, ground rice, and hide glue was referenced as being the whitewash used on the White House, and it continued to get published until the 1880s (this was Recipe 3 tested for this thesis).<sup>61</sup> Two additional recipes which were published for the first time in the 1860s call for the use of lime, salt, and zinc sulfate with one of the recipes also adding in milk. These two were copied over and over again in different books until about 1920.

On the other hand, many recipes were only published once. It is difficult to say how certain recipes became more popular than others, but theoretically, the more durable and effective mixtures should have been replicated the most. That is one of the questions the testing in this thesis attempted to answer. With the supposed White House whitewash though, it may have been copied so frequently because of its association with the prestigious building rather than its performance. Regardless of the reasons, some recipes survived much longer than others, and some were likely never written down at all. It would be impossible to look back and completely understand the reasons why, but doing testing of historic limewash recipes is the way in which this thesis begins to look at it.

In order to better understand the purpose of testing, it is helpful to examine the additives used in the recipes. There is a wide variety of ingredients published in 19th-century recipes, but not all of these were commonly mentioned. For the purposes of this thesis, “commonly” refers to an additive that was published at least five times, based on the compiled spreadsheet of recipes.

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<sup>61</sup> John Agg Papers. 1830. David M. Rubenstein Rare Book & Manuscript Library, Duke University. Cited in Johansen, E.L.A. 1999. ‘Deterioration of Gneiss Due to Limewash at Eastern State Penitentiary’. Thesis. University of Pennsylvania.

The spreadsheet is not exhaustive by any means, but it is representative of many different sources and recipes. The following the ingredients were deemed common, from most to least: salt, whiting, zinc sulfate, hide glue, milk, ground rice (or rice flour), linseed oil, alum, sugar/molasses, and sand. Some of these were particularly frequent because of the repeated recipes, but they were still counted as “commonly” used. By far, salt was the most common additive, appearing in numerous different recipes, as well as repeated ones. Alternatively there were other ingredients only mentioned a few times; these include rosin, wood ashes, flour, blood, beer grounds, bluing, tallow, Burgundy pitch, cement, and soap. Of course, these ingredients could have been frequently used and learned by oral or local tradition, but that cannot be known for certain without testing of existing historic limewash. Since the former list of additives was surely more widely circulated, those are the ones that are focused on in this thesis. Each one of those, except for linseed oil and sand, are tested in one or more recipes, listed in Section 5.2. The reasons that linseed oil and sand were not included are twofold. First, linseed oil and sand were mentioned less frequently than many of the other additives. Second, and more importantly, linseed oil and sand as additives both begin to blur the lines between limewash and oil paint and plaster, respectively. Those materials may very well help create a good, durable wall coating, but it is potentially too far divorced from a traditional limewash for the purposes of this thesis. Therefore, salt, whiting, zinc sulfate, hide glue, milk, ground rice, alum, and sugar are the distinct additives used in testing.

#### 4.2 Historic claims

While there are numerous unanswered questions about 19th-century limewash use, what people believed the additives would do was not one of them. Some sources explicitly state what

qualities of a limewash that certain additives were intended to alter. The following list details these claims:

**Alum** added to a lime whitewash prevents it rubbing off. An ounce to the gallon is sufficient. **Molasses** renders the lime more soluble and causes it to penetrate the wood or plaster surface; a pint of molasses to 5 gallons whitewash is sufficient.

- Walker, P.H. 1913. 'The Use of Paint on the Farm'. In *Eighteenth Biennial Report*, 142-156. Topeka: Kansas Department of Agriculture.

For lime washing, one pound of **sugar** to sixteen gallons of water will make it adhere splendidly. Drops of whitewash made with water so mixed, if they fall on the floor or window, or an iron plate, cannot be washed off.

'Sugar and Mortar'. *The Manufacturer and Builder*, **22**(3): 57, 1890.

I am enabled to certify the efficacy of **marine salt** in fixing whitewash made of lime. ... The whitewash produced by it is very permanent, does not crack, not come off upon one's hands or clothes. The experiment was made only on wood.

- Levavasseur, Gen. 1811. 'Method of rendering Whitewash made with Lime durable', *The Repertory of Arts, Manufactures, and Agriculture*, vol. 2. London: Nichols.

The best method for making a whitewash for outdoor exposure is to slack half a bushel of lime in a barrel, add one pound of common **salt**, half a pound of **sulphate of zinc**, and a gallon of sweet **milk**.

- I.R. Butts. *The Tinman's Manual and Builder's and Mechanic's Handbook*, fourth edition. Boston: I.R. Butts Co. 1867, 86.

Lime slacked with a solution of **salt** in water, and thinned with **skimmed milk**, from which all cream has been taken, makes a permanent whitewash for out-door work and it is said, renders the wood incombustible

- Shaker Manifesto, 1881

Lassiter, William. *Shaker Recipes and Formulas*. New York: Bonanza, 1978. 248-9.

Wash that Won't Rub Off. – The following is recommended. Mix up half a pailful of lime and water; take half a pint of **flour** and make a starch paste of it, and pour it into the whitewash while hot. Stir it well and make it ready for use.

- Dr. Geo. Blakelee. *Blakelee's Industrial Encyclopedia*, New York: Universal Information Exchange, Publishers, 1887, 162-167.

Substitute for White-Lead.—Take one bushel of unslacked lime, and slack it with cold water. When slacked, add twenty pounds of **Spanish whiting**, seventeen pounds of **salt**, and twelve pounds of **sugar**. Strain this mixture through a wire-sieve, and it will be fit for use, after reducing it with water. This is intended for the outside of buildings, or where it is exposed to the weather.

- E. G. Storke, *The Family and Householder's Guide* [etc.], Auburn, NY: The Auburn Publishing Company, c.1859.



A first-class wash for external work is formed by taking a shovelful of good lime, and slake it in about a quart of warm **blood**, fresh from the slaughter house. Add to this a sufficient quantity of **beer grounds**, or **stale beer** and **skim milk**, in equal parts, boiled together, to fill with the lime and blood an ordinary pail. ... This wash will stand the weather better than oil paint.

- Frederick Thomas Hodgson, *Plaster and Plastering. Mortars and Cements, How to Make, and How to Use*, New York: The Industrial Publication Company, 1883.

## 5. TESTING DESIGN

This testing plan was designed to evaluate some basic limewash properties so that the results could be compared to historical claims. The plan was formulated after a visit to NCPTT in Natchitoches, Louisiana where the author was able to see how they carried out their limewash testing. NCPTT studied the durability of limewashes with different types of lime and recipes using tests for abrasion, adhesion, and QUV artificial weathering. Their testing model is similar to the one chosen for this thesis.

### 5.1 Tests

Three ASTM tests were chosen: adhesion (ASTM D3359), chalking resistance (ASTM D4214), and water-vapor transmission (ASTM E96). The abrasion and chalking resistance tests help to compare historic claims about limewash additives to actual measurable parameters. For example, looking at the claims stated in Chapter 4, the *Manufacturer and Builder* article from 1890 states that the addition of sugar will make “a limewash adhere splendidly” and that it cannot be washed off.<sup>62</sup> ASTM D3359, the adhesion test, was able to test this claim compared to a pure limewash and other recipes. The full discussion of testing results to verify or potentially discredit these claims is laid out in Chapter 8. To simplify this information, a chart is helpful. The following chart distills down the historic claims from Section 4.2 and lists which ASTM test best evaluates the claims. The second half of the chart lists other claims not tested for this thesis but could be studied by other researchers.

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<sup>62</sup> ‘Sugar and Mortar’, 57.

Additive	Function/Claim	Test
Alum	Prevents rubbing off	Chalking resistance
Sugar/molasses	Penetrate surface and adhere well	Adhesion
Salt	Wash will not crack or come off in one's hands	Chalking resistance and adhesion
Salt, zinc sulfate, and milk	For outdoor work	Adhesion, water-vapor transmission
Ground rice, hide glue, whiting, salt	No specific function listed	N/A
<b>Claims not tested for this thesis:</b>		
Salt with milk	Renders wood incombustible	None
Flour	Won't rub off	Chalking resistance
Spanish whiting, salt, sugar	Substitute for white lead, good for outdoor exposure	Adhesion, water-vapor transmission, artificial or natural weathering
Blood, beer grounds or stale beer, and skim milk	Better than oil paint for outdoor work	Adhesion, water-vapor transmission, artificial or natural weathering

The water-vapor transmission test was not intended to answer any specific historic claims but rather show how additives affect the breathability of a limewash. Since breathability is one of the most commonly cited benefits of limewash this was an important quality to assess. Despite this, there have not been many studies about the effects of additives or multiple coats on breathability.<sup>63</sup> These tests allow for a better understanding of breathability by comparing a pure limewash to recipes with additives as well as the effects of multiple layers.

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<sup>63</sup> One study by J. Boxall and P.M. Trotman, "Limewash: Water Vapour Permeability" in the *Journal of Oil and Colour Chemists' Association*, vol. 79, issue 3, tests water vapor transmission of limewashes made of lime and linseed oil, lime and tallow, and lime and milk using two coats each. However, none of these recipes were tested in this thesis. Therefore, the ASTM E96 tests for this thesis use different recipes and number of coats than Boxall and Trotman.

## 5.2 Limewash samples

For the adhesion and chalking tests, five historic recipes and one control pure limewash were chosen. The recipes are listed below:

Recipe:	Composition:	Source:
1	Lime and water	Control
2	Lime, water, salt, milk, zinc sulfate	Multiple <sup>64</sup>
3	Lime, water, salt, whiting, ground rice, hide glue	Multiple <sup>65</sup>
4	Lime, water, salt	The Household Painter, 1923
5	Lime, water, sugar	Manufacturer and Builder, 1890
6	Lime, water, alum	National Painter's Magazine, 1908

The first two recipes were chosen because they were the most frequently published recipes from the 19th century. These were continuously published in different sources over a period of 50 years each.<sup>66</sup> The other three recipes contain commonly published additives: salt, sugar, and alum. However, no identical recipes containing these ingredients were written over and over again as with Recipes 1 and 2; they were used in a number of different recipes throughout the 19th century. A wide variety of other ingredients were mentioned in different sources,<sup>67</sup> but because they were infrequently mentioned, they were not included in this testing plan.

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<sup>64</sup> The Tinman's Manual and Builder's and Mechanic's Handbook, 1867; Workshop Receipts for the Use of manufacturers, Mechanics, and Scientific, 1879; Plaster and Plastering, 1883; Blakelee's Industrial Encyclopedia, 1883; Kansas Dept. of Agriculture, 1913.

<sup>65</sup> John Agg Family papers, 1830; Scientific American, 1843; Southern Planter, 1843; American Penny Magazine and Family Newspaper, 1845; Shaker Manifesto, 1881; Plaster and Plastering, 1883.

<sup>66</sup> Recipe 1 appears in sources from 1830-1883, while Recipe 2 appears from 1867 to 1913.

<sup>67</sup> These are detailed in Chapter 3.2.

### 5.3 Substrate

Because limewash can be used on most porous surfaces, the substrate used for these tests could have been a number of different materials. Brick was ultimately chosen because it is a readily available material that limewash was known to have been used on. Old Mill Brick Company brick thins were used because they are easier to work with than regular brick but still yield the same results. Some of the bricks had a smooth, less porous, modern brick surface while other bricks had a rougher, more porous surface more closely mirroring historic brick. These two types of surfaces allowed for comparison of the test results.

Performing the three tests on wood would have also been a viable option; if this thesis were to be continued, testing the same plan on wood samples would be recommended. NCPTT performed their tests on both brick and wood, finding that none of the limewashes tested on wood were as durable as those tested on brick. It would be interesting to learn how the recipes tested in this thesis perform on wood.

### 5.4 Number of coats

There is no one rule as to the number of coats of limewash that should be applied to a wall or structure. Many sources state 3-5 coats of limewash are adequate for the first application.<sup>68,69</sup> However, as we know from written accounts, some homeowners applied a new coat of limewash on a yearly basis, either painting over all of the old coats or scraping others off prior to limewashing.<sup>70</sup> Therefore, the number of coats is highly variable. For this study, 3 coats and 5 coats were chosen to represent a possible first application of limewash. Each of the recipes was therefore applied to the brick samples in coats of 3 and 5.

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<sup>68</sup> Storke, 1859.

<sup>69</sup> Mold and Godbey, 9.

<sup>70</sup> Notter, J. Lane. 1908. *The Theory and Practice of Hygiene (Notter and Firth) Revised and Largely Rewritten by R.H. Firth*. London: J&A Churchill, 427.

## 5.5 Additives

Finding additives in today's world that would accurately represent 19th-century ingredients was a challenge. Most of the additives cannot correspond exactly to what would have been used by people mixing limewash, but these were the best possible approximations available to the author.

Additive	Used in testing	Historically
Salt	Coarse NaCl	Salt could have come from rock salt or boiling natural brine (from springs or sea water). <sup>71</sup> Rock salt is predominantly NaCl with a small amount of other salts. Salt from natural brine is variable. <sup>72</sup>
Milk	Ronnybrook Milk (non-homogenized)	Unpasteurized milk would most likely have been used, but this is not a commonly available ingredient today.
Zinc sulfate	Zinc sulfate (ZnSO <sub>4</sub> )	Zinc sulfate (ZnSO <sub>4</sub> )
Alum	Alum (potassium aluminum sulfate)	Alum (potassium aluminum sulfate)
Ground rice	White rice ground with a mortar and pestle	Multiple rice varieties would have been available. <sup>73,74</sup> No recipes specify a type of rice but some others do say rice flour.
Hide glue	Ground hide glue	Hide glue was made by boiling the skins, hoofs, etc. of animals. <sup>75</sup>

<sup>71</sup> Bertram, Bruce M. 2006. 'Salt Mining Technology'. In *The American Midwest: An Interpretive Encyclopedia*, Cayton, Andrew R.L., Sisson, Richard, and Zacher, Chris (eds.) 1468. Bloomington, IN: Indiana University Press.

<sup>72</sup> Ryzhenko, B.N. and Cherkasova, E.V. 2012. 'Chemical Composition of Natural Waters and Brines as a Result of Hydrogeochemical Processes in Water - Rock - Gas Systems', *Geochemistry International*, **50**(13): 1101-1150.

<sup>73</sup> About half of the rice consumed in the U.S. was produced there and the other half was imported by the end of the 19<sup>th</sup> century. The different types would have included gold seed (long- and short-grained), white rice (short-grained), and brown rice. White rice was chosen for testing because it was cited as being one of the more inexpensive rice choices in the 19<sup>th</sup> century. Austin, Amory. 1893. *Rice: Its Cultivation, Production, and Distribution in the United States and Foreign Countries*. Washington, D.C.: Government Printing Office, 15-24.

<sup>74</sup> Knapp, S.A. 1899. *The Present Status of Rice Culture in the United States*. Washington, D.C.: Government Printing Office.

<sup>75</sup> Rideal, Samuel. 1900. *Glue and Glue Testing*. London: Scott, Greenwood and Co., 1.

Sugar	Raw sugar	Multiple types of sugar would have been available. <sup>76</sup> No specific type of sugar was specified for this limewash, so raw sugar was used since this would have been the least expensive option.
Whiting	Whiting (calcium carbonate)	Whiting (calcium carbonate), although it may have contained traces of magnesium carbonate, silica, iron, and alumina. It was not likely to be adulterated though, because it was inexpensive. <sup>77</sup>

## 5.6 Brushes

Historically, the type of brush used for limewashing was of a specific type. “Whitewash brushes” were often referenced in articles and books. These brushes were described as “made of long bristles fastened to a flat wooden handle.”<sup>78</sup> The brushes could be quite large, up to nine inches wide and many bristles deep. Whitewash brushes could have been comprised of animal hair or plant fibers, depending on when and where they were made.<sup>79</sup> Today, the term “whitewash brush” largely means the same thing, but they are mostly made of synthetic or plant fibers. Unfortunately, a traditional brush was found to be too large for applying limewash to the brick samples in these tests. Instead, smaller brushes with a large amount of bristles made of goat hair were chosen for testing.

<sup>76</sup> Brown sugar, white sugar, molasses, raw sugar, and sugar in various states of refinement would have been available in 19<sup>th</sup>-century America. More refined sugar was more expensive than less refined sugar, so it is likely that for a limewash, a less expensive option would have been used. Mescher, Virginia. 2005. “How Sweet it is!” A History of Sugar and Sugar Refining in the United States’. Accessed February 21, 2015. [http://www.raggedsoldier.com/sugar\\_history.pdf](http://www.raggedsoldier.com/sugar_history.pdf).

<sup>77</sup> Ellis, George H. 1898. *Analysis of White Paints: A Collection of Notes on the Chemical Analysis of White and Tinted Paints*. Evanston, IL: The Technica Press.

<sup>78</sup> ‘How Brushes are Made’, *The Manufacturer and Builder*, **23**(12): 267, 1891.

<sup>79</sup> Foss, Walter D. 1922. ‘How the Brush Industry Has Developed During the Lifetime of One of Its Pioneers’, *The Painters Magazine*, **49**(12): 42.

### 5.7 Number of samples

The tests for adhesion and chalking resistance were performed in triplicate, resulting in 36 samples. Recipes 1-6 were painted on three thin bricks in both 3 and 5 coats. ASTM D3359 and D4214 tests were performed on opposite sides of each thin brick sample.

ASTM E96 was performed with 5 samples for limewash Recipes 1-3 (also each in 3 and 5 coats), resulting in a total of 30 samples. Five uncoated brick samples were also tested, resulting in a total of 35 water-vapor transmission dish assemblies. In order to create these samples, cores were taken from the thin bricks.

The number of samples is laid out in the following charts:

Sample#	Recipe-Coats	Test	Test
1	1-3	Adhesion	Chalking Resistance
2	1-3	Adhesion	Chalking Resistance
3	1-3	Adhesion	Chalking Resistance
4	1-5	Adhesion	Chalking Resistance
5	1-5	Adhesion	Chalking Resistance
6	1-5	Adhesion	Chalking Resistance
7	2-3	Adhesion	Chalking Resistance
8	2-3	Adhesion	Chalking Resistance
9	2-3	Adhesion	Chalking Resistance
10	2-5	Adhesion	Chalking Resistance
11	2-5	Adhesion	Chalking Resistance
12	2-5	Adhesion	Chalking Resistance
13	3-3	Adhesion	Chalking Resistance
14	3-3	Adhesion	Chalking Resistance
15	3-3	Adhesion	Chalking Resistance
16	3-5	Adhesion	Chalking Resistance
17	3-5	Adhesion	Chalking Resistance



18	3-5	Adhesion	Chalking Resistance
19	4-3	Adhesion	Chalking Resistance
20	4-3	Adhesion	Chalking Resistance
21	4-3	Adhesion	Chalking Resistance
22	4-5	Adhesion	Chalking Resistance
23	4-5	Adhesion	Chalking Resistance
24	4-5	Adhesion	Chalking Resistance
25	5-3	Adhesion	Chalking Resistance
26	5-3	Adhesion	Chalking Resistance
27	5-3	Adhesion	Chalking Resistance
28	5-5	Adhesion	Chalking Resistance
29	5-5	Adhesion	Chalking Resistance
30	5-5	Adhesion	Chalking Resistance
31	6-3	Adhesion	Chalking Resistance
32	6-3	Adhesion	Chalking Resistance
33	6-3	Adhesion	Chalking Resistance
34	6-5	Adhesion	Chalking Resistance
35	6-5	Adhesion	Chalking Resistance
36	6-5	Adhesion	Chalking Resistance

Sample#	Recipe-Coats	Test
37	1-3	Water-vapor transmission
38	1-3	Water-vapor transmission
39	1-3	Water-vapor transmission
40	1-3	Water-vapor transmission
41	1-3	Water-vapor transmission
42	1-5	Water-vapor transmission
43	1-5	Water-vapor transmission
44	1-5	Water-vapor transmission
45	1-5	Water-vapor transmission

46	1-5	Water-vapor transmission
47	2-3	Water-vapor transmission
48	2-3	Water-vapor transmission
49	2-3	Water-vapor transmission
50	2-3	Water-vapor transmission
51	2-3	Water-vapor transmission
52	2-5	Water-vapor transmission
53	2-5	Water-vapor transmission
54	2-5	Water-vapor transmission
55	2-5	Water-vapor transmission
56	2-5	Water-vapor transmission
57	3-3	Water-vapor transmission
58	3-3	Water-vapor transmission
59	3-3	Water-vapor transmission
60	3-3	Water-vapor transmission
61	3-3	Water-vapor transmission
62	3-5	Water-vapor transmission
63	3-5	Water-vapor transmission
64	3-5	Water-vapor transmission
65	3-5	Water-vapor transmission
66	3-5	Water-vapor transmission

## 6. TEST METHODS

### 6.1 Pre-testing

Before beginning actual testing, a pre-test sample was prepared in order to practice mixing limewash and to perform the adhesion and chalking resistance tests. To make the limewash, the proportions suggested in Peter Mold and Richard Godbey's "Limewash: Compatible Covering for Masonry and Stucco" were followed. They suggest a ratio of 15-20% lime solids and 80-85% water.<sup>80</sup> For this test, approximately 20% lime and 80% water was used by mixing 170 g Graymont Niagara Lime Putty with 260 g water. That mixture is 19.76% lime by weight, because lime putty contains approximately 50% lime and 50% water.<sup>81</sup> The resulting limewash appeared too thick, based on historic descriptions that the wash should be the consistency of whole milk or cream.<sup>82</sup> By adding 70g more water, the limewash became a whole milk consistency. The resulting limewash then contained 17% lime. Therefore, this approximate ratio was decided upon for the control limewash. Many of the historic recipes do not specify the amount of water (only the lime and additives are given). When mixing these recipes, the basic 17% lime, 83% water formula was followed.

The sample brick was coated with three layers of limewash, applied 24 hours apart. Between coats, the brick was misted with water to keep the limewash damp and prolong curing. Allowing the limewash to cure for a longer amount of time is important so that it does not "dry out" without fully carbonating. Below, these are photos taken 24 hours after each coat was applied (and immediately prior to the subsequent coat).

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<sup>80</sup> Mold and Godbey, 4.

<sup>81</sup> Todd D. File, Graymont Account Manager confirmed this to be true for Graymont Niagara Lime Putty as well.

<sup>82</sup> Mold and Godbey also state that adjustments may need to be made since lime putties can vary.

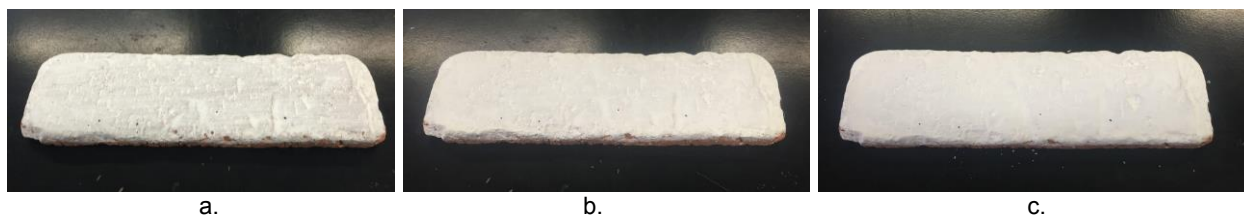


Figure 4. Test sample brick after a) 1 coat, b) 2 coats, and c) 3 coats of pure limewash

Then, 48 hours after the third coat was applied, ASTM D3359 and D4214 were performed in order to practice for the actual testing. Since these tests were done in isolation, no data for comparative analysis was obtained. However, the tests could be compared to the ASTM standards for classification.

The tape adhesion test was performed using the cross-hatching method according to ASTM D3359. Six lines, 2mm apart and 20mm long were cut vertically then another 6 lines were cut horizontally to hatch the first set of lines. Each cut was made to reveal the brick substrate. The area was then brushed off to remove any pieces of flaking limewash. Finally, a piece of tape<sup>83</sup> was applied over the cross-hatch area for 90 seconds then removed at almost a 180 degree angle. The result was compared to the “Classification of Adhesion Test Results” chart in the ASTM standard. The coating appeared to be about 35% detached, which is classified as 1B or 2B. The detachment was predominantly between the 1st and 2nd coats.

To perform the chalking resistance test according to ASTM D4214, Test Method D was used. A piece of pressure sensitive tape was laid on the limewashed brick, then removed. This piece of tape was compared to “Photographic Reference Standard No. 2.” The result most closely matched Category 4. However, the limewash appeared to come off on the tape in flakes rather than an allover chalking appearance as shown in the ASTM reference standards. Even though

<sup>83</sup> For this test, Scotch tape was used. For the actual testing, M.E. Taylor’s Semicro CHT tape was used. This is the recommended replacement tape for Permacel 99, which was the ASTM specified tape but is no longer manufactured.

these results alone do not yield salient data, performing both this test and the adhesion test was useful for practicing the test methods.

Additionally, before testing could begin, the brick samples were prepared by labeling them according to the recipe number, number of coats, and triplicate number. Thirty-six brick samples were labeled. Additionally, thirty-five samples for water-vapor transmission were created using a 45mm drill core. Once these steps were completed, testing began.



Figure 5. Drilling 45mm cores of brick

## 5.2 Painting samples and observations



Figure 6. Painting limewash onto brick samples

Each of the brick samples and test cores were painted with limewashes mixed from the historic recipes listed in Section 5.2 and detailed in Appendix A. The appendix gives the conversion of recipe proportions to make a smaller amount of each limewash and the precise amount of each ingredient used. This section details how each recipe was mixed and initial observations about the coating.

## Recipe 1

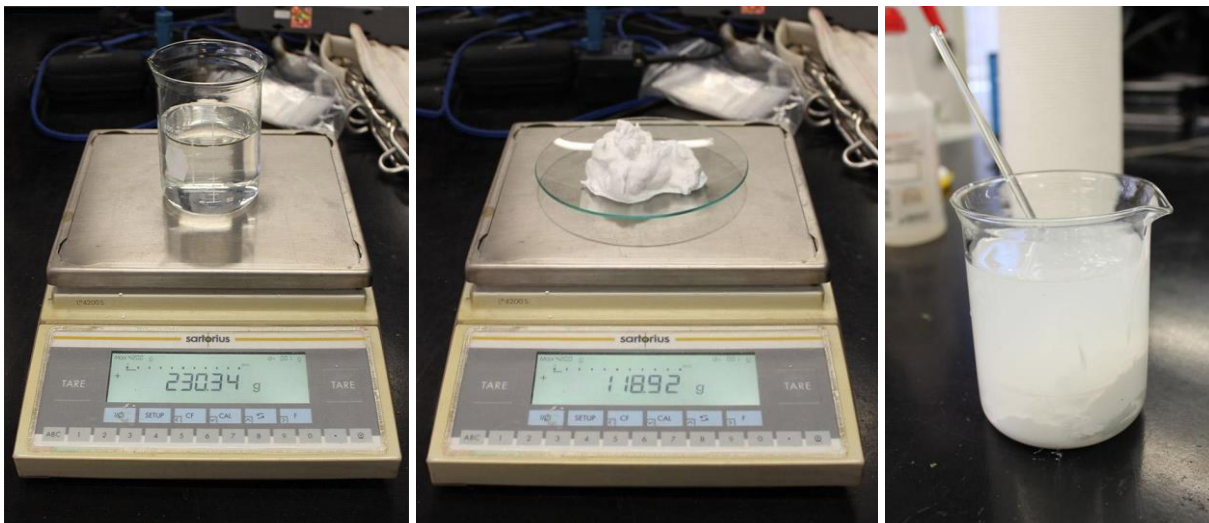


Figure 7. Weighing ingredients to make Recipe 1

Recipe 1 was the control wash of lime and water. To create the 17% lime percentage discussed in Section 7.1, 119g of lime putty was used with 230g of water. Because the lime putty is approximately 50/50 lime to water, this mixture is actually 59.5g lime to 289.5g water, a 17:83 lime to water ratio. According to historic recipes as well as Mold and Godbey's study, limewash should have a viscosity similar to whole milk or cream. The limewash was poured from one beaker into another to verify that the 17:83 ratio did result in this consistency.

The wash was brushed onto the samples horizontally for the first coat, vertically for the second coat, and then alternating for the remaining coats. The coats were applied 24 hours apart and misted with water before each application, as



Figure 8. Pouring to verify whole milk to cream-like consistency

was the case with all of the recipes.<sup>84</sup> In terms of workability, this recipe brushed onto the brick samples easily.

Bricks painted with Recipe 1 appeared to cure more quickly than all of the other recipes. This was noted because while limewash looks fairly transparent when first applied, it turns white and opaque as it cures. Recipe 1 turned white at a faster rate than Recipes 2-6. As a whole, this recipe appeared the most aesthetically pleasing, yielding an even, bright white coating. There was little change in appearance (opaqueness or color) from coats 3 to 5. The following photos show bricks painted with Recipe 1 after each coat.

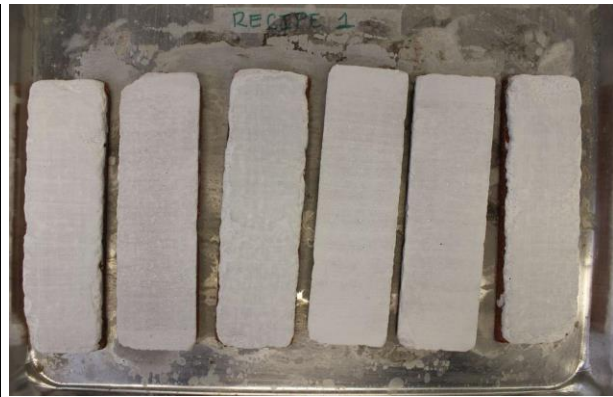
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<sup>84</sup> These techniques were learned during the author's summer internship with the NPS through correspondence with practitioners and painting experts.

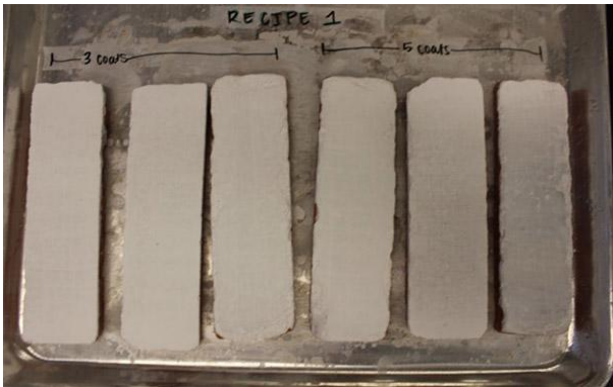




a.



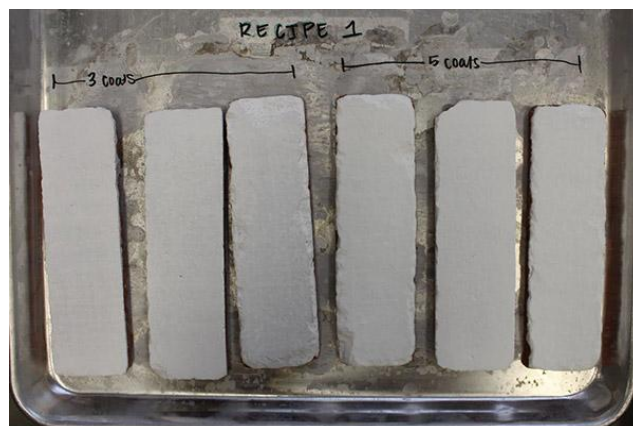
b.



c.



d.



e.

Figure 9. Recipe 1 after a) one, b) two, c) three, d) four, and e) five coats. Only the three bricks on the right of d. and e. received four and five coats. The three left brick have three coats each.



## Recipe 2

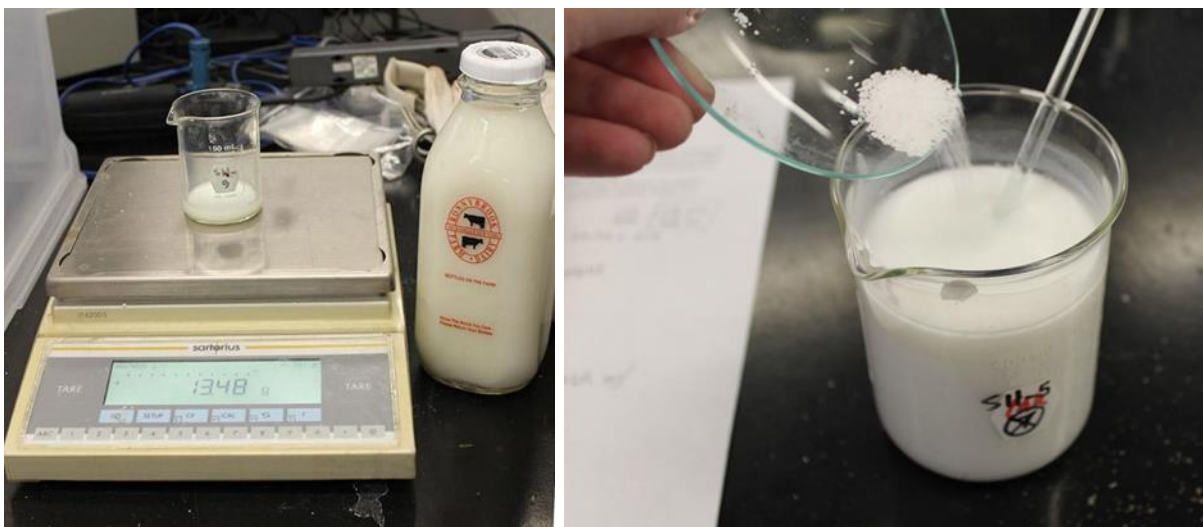


Figure 10. Measuring and mixing ingredients for Recipe 2

Recipe 2 consisted of lime, water, salt, zinc sulfate, and milk. It was mixed with the same amount of lime as Recipe 1, but with less water to make up for the addition of milk. This resulted in the same whole milk consistency as the control limewash. Recipe 2 did not brush on as easily as Recipe 1, because some of the lime particles seemed to become matted in the brush's bristles. Ultimately, this recipe did not provide as much hiding power as the control wash in the first few coats; the substrate showed through more and brush strokes were more visible. Also, the coating was closer to a cream color than the control wash. The following photos illustrate this contrast.

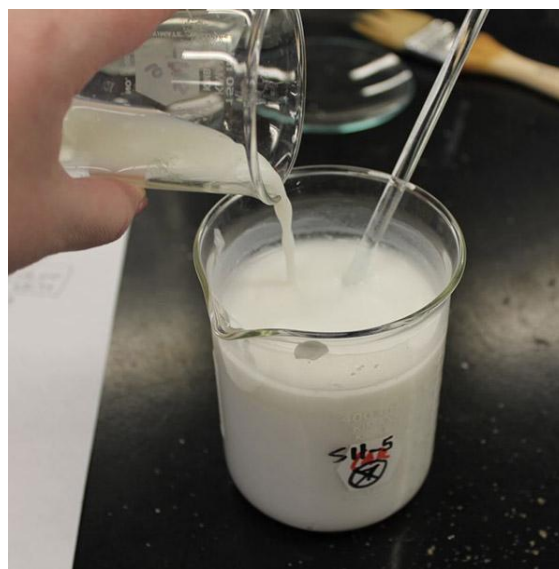


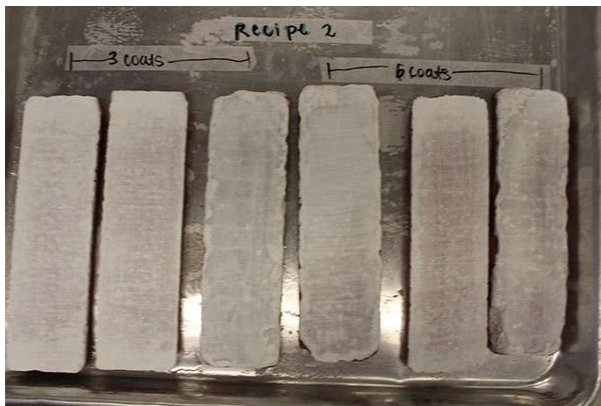
Figure 11. Pouring milk into limewash to create Recipe 2



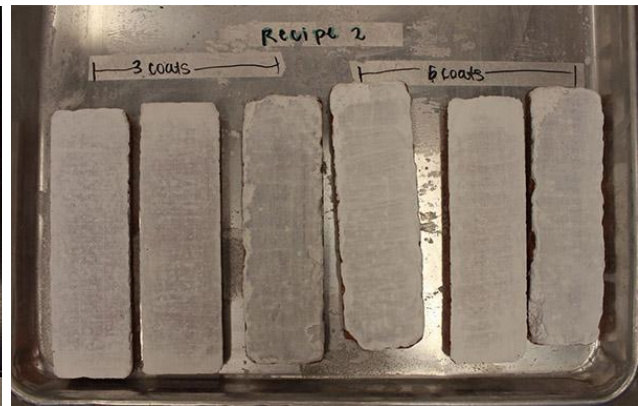
a.



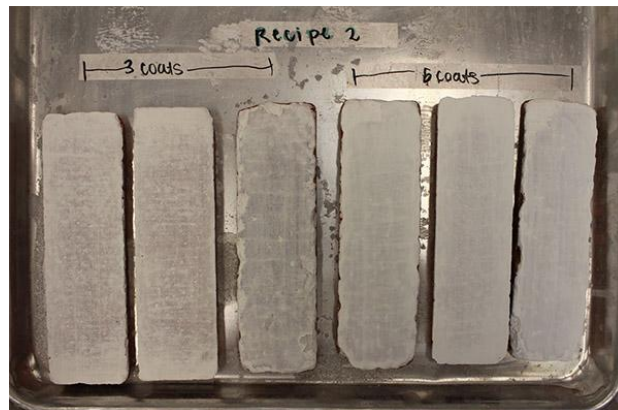
b.



c.



d.



e.

Figure 12. Recipe 2 after a) one, b) two, c) three, d) four, and e) five coats. Only the three bricks on the right of d. and e. received four and five coats. The three left brick have three coats each.

### Recipe 3



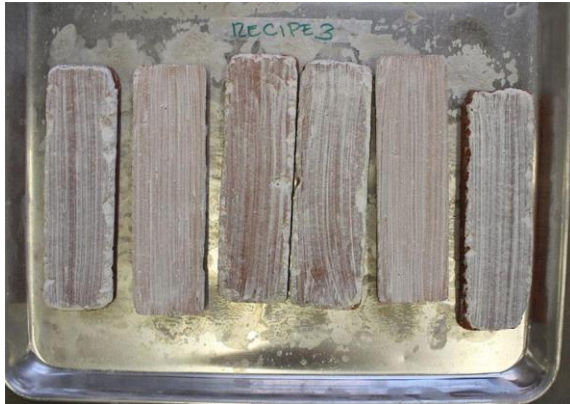
Figure 13. Mixing and measuring the ingredients for Recipe 3 and grinding rice with mortar and pestle

Recipe 3 was by far the most difficult to mix and use. This recipe required many steps and ingredients (lime, water, salt, ground rice, whiting, and hide glue), and unlike the other recipes, it needed to be put on hot. To begin the process, rice was ground with a mortar and pestle into a fine powder. Water was then added to the ground rice and boiled to a thin paste. In addition to the rice paste, the recipe required that the salt be dissolved in hot water before being

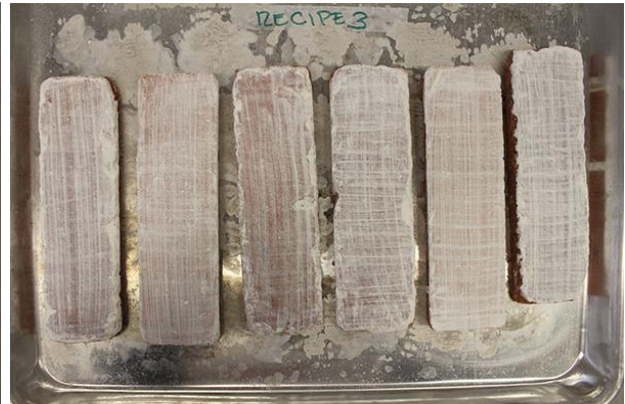
added to the wash. The hide glue was also dissolved in water before being added. Coordinating all of the steps and additions was both complicated and challenging, but it eventually came to the proper consistency. Recipe 3 was kept on a hot plate until needed, as the historic source says to keep it on a portable furnace.

One of the first things noted about this recipe was that the particles did not stay in suspension as long as the other recipes; they began to settle to the bottom much more quickly. Applying Recipe 3 also presented a challenge. The particles clung to the brush bristles, which made it difficult to apply, and it did not go on smoothly. The brushstrokes were evident and the substrate showed through the wash in many places. A truly opaque coating was not achieved until the 5th coat. In terms of workability and aesthetics, Recipe 3 seemed to perform the worst even though it was commonly cited in many historic sources.

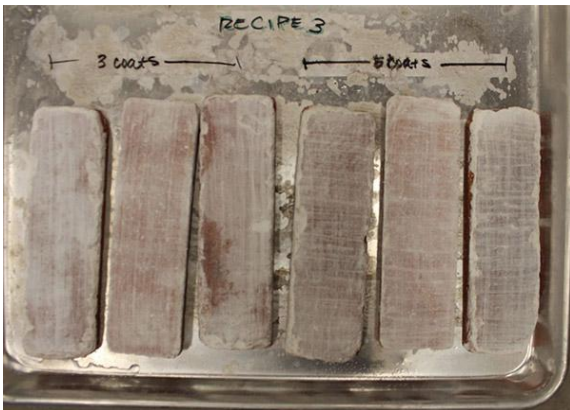




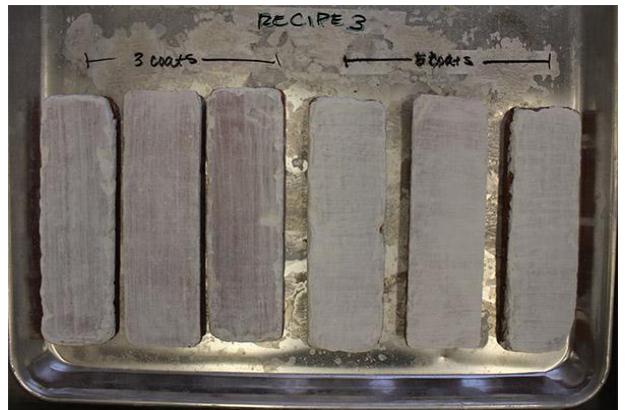
a.



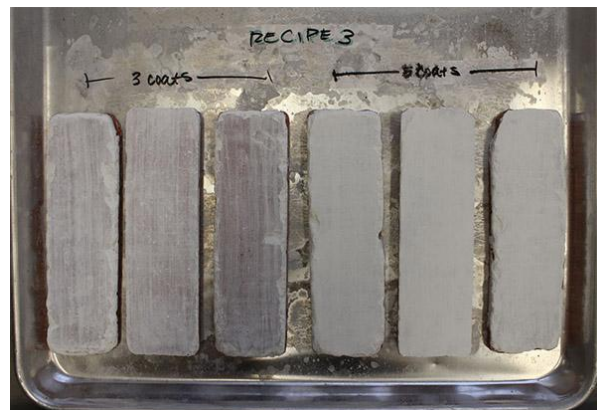
b.



c.



d.



e.

Figure 14. Recipe 3 after a) one, b) two, c) three, d) four, and e) five coats. Only the three bricks on the right of d. and e. received four and five coats. The three left brick have three coats each.

## Recipe 4

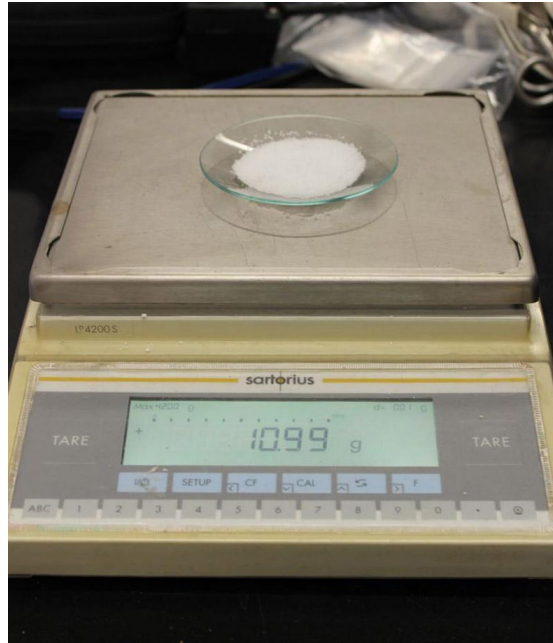
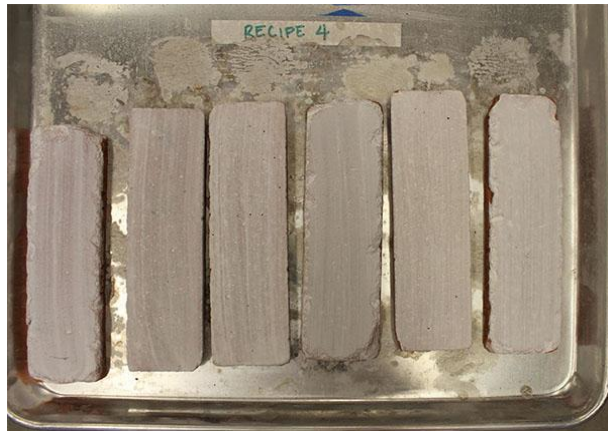


Figure 15. Salt used to make Recipe 4

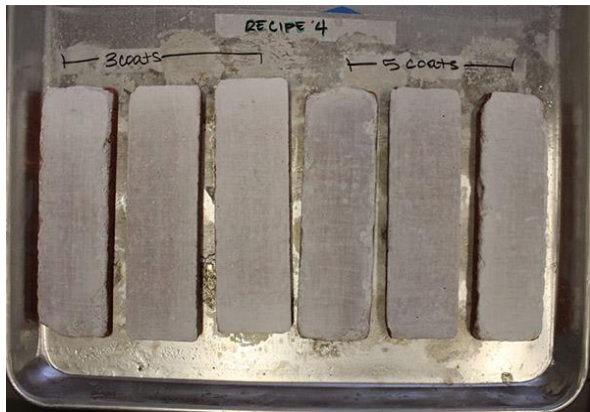
Recipe 4 was mixed almost identically to Recipe 1 but also added in salt. The same amounts of lime and water were used as well as 11g of salt. This recipe brushed on the easiest of all mixtures, and provided good hiding power but still not as good as Recipe 1. Aside from the control wash, Recipe 4 cured the most quickly of all the recipes.



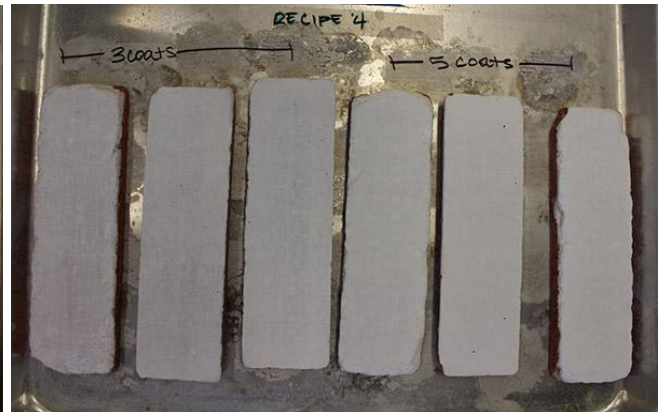
a.



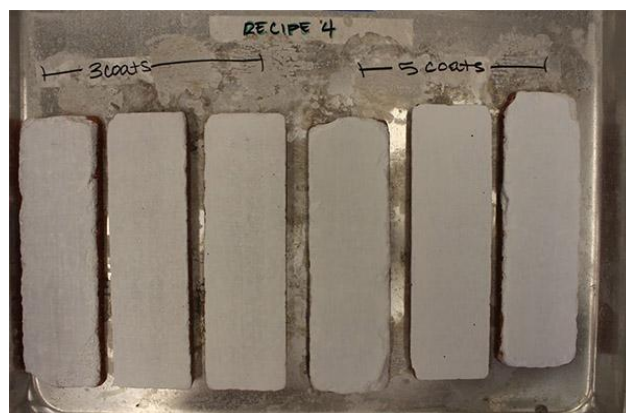
b.



c.



d.



e.

Figure 16. Recipe 4 after a) one, b) two, c) three, d) four, and e) five coats. Only the three bricks on the right of d. and e. received four and five coats. The three left brick have three coats each.



## Recipe 5

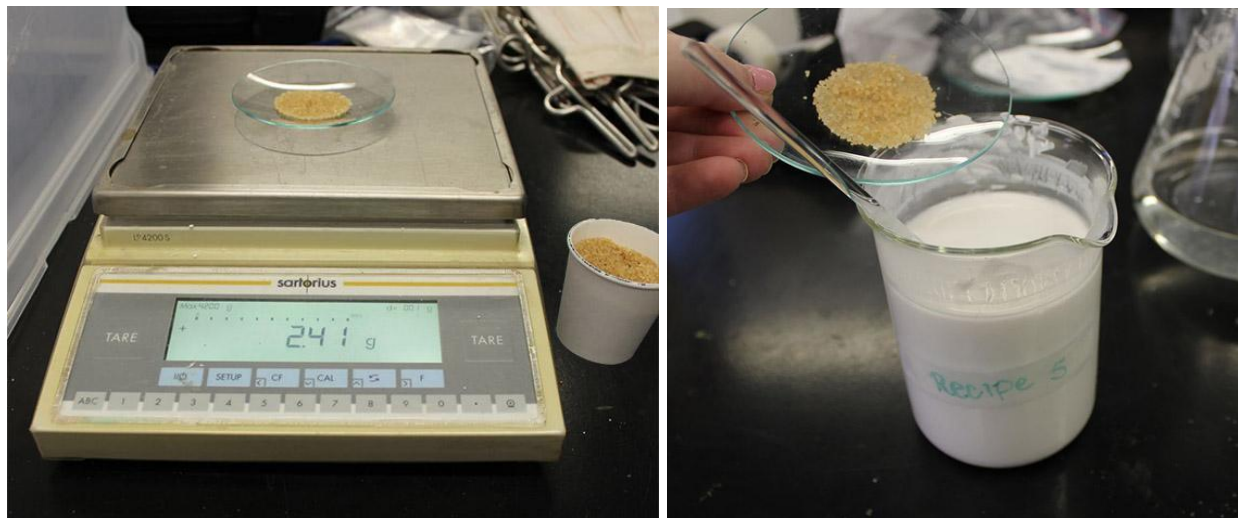


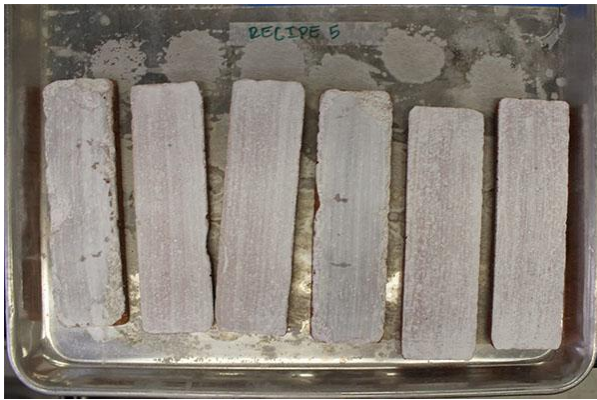
Figure 17. Measuring and adding sugar to limewash mixture to make Recipe 5

Recipe 5 was prepared the same way as Recipe 1 and 4 with the same amounts of lime and water but with 2.41g sugar. Interestingly, this mixture seemed thinner than those other two recipes even with the same proportions. The Kansas State Department of Agriculture stated that molasses renders lime more soluble, so perhaps this is the reason that the sugar recipe was thinner than Recipes 1 and 4.<sup>85</sup> After the first coat of Recipe 5 cured, two of the bricks had unexplained spots without limewash. These spots were covered up by subsequent coats, but the reason for the spots is not known, and they can be seen in Figure 18a. Even though the limewash was able to cover those spots, Recipe 5 did not provide a great amount of hiding power and was splotchy in some parts. This can be seen in the following photos.

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<sup>85</sup> Walker, 142-156.

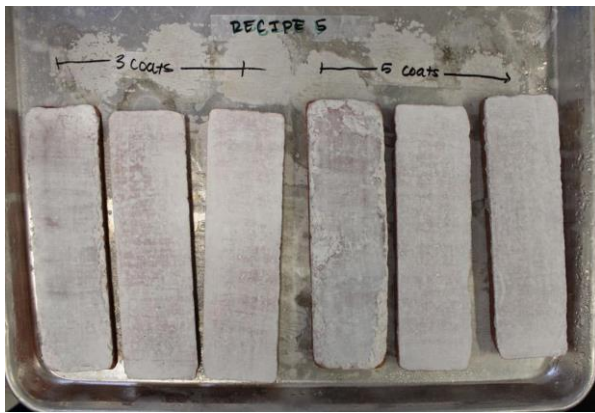




a.



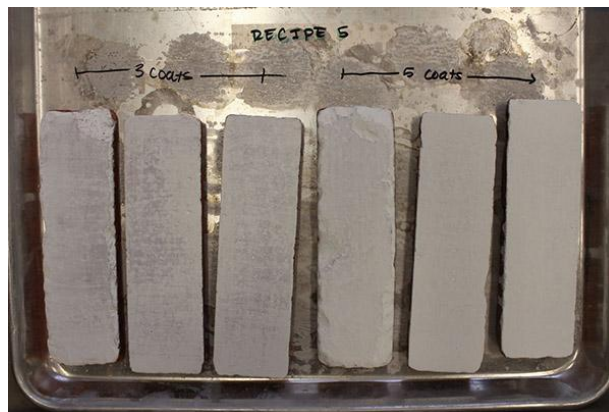
b.



c.



d.



e.

Figure 18. Recipe 5 after a) one, b) two, c) three, d) four, and e) five coats. Only the three bricks on the right of d. and e. received four and five coats. The three left brick have three coats each.

## Recipe 6

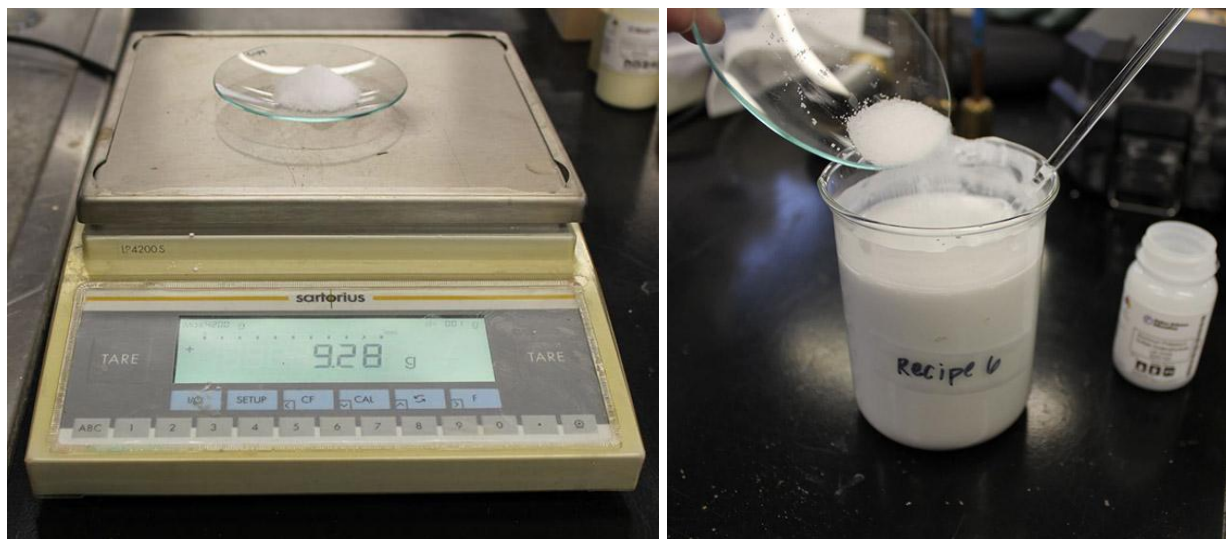
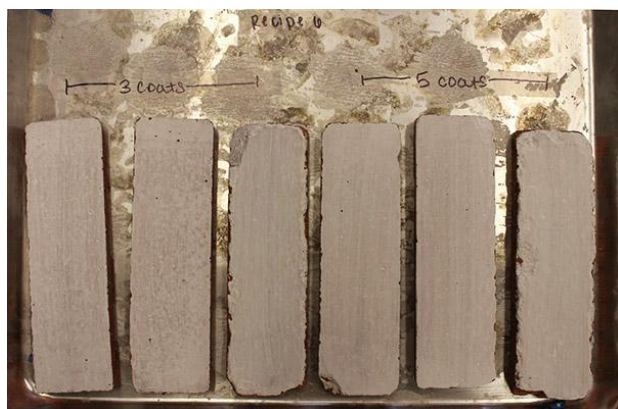
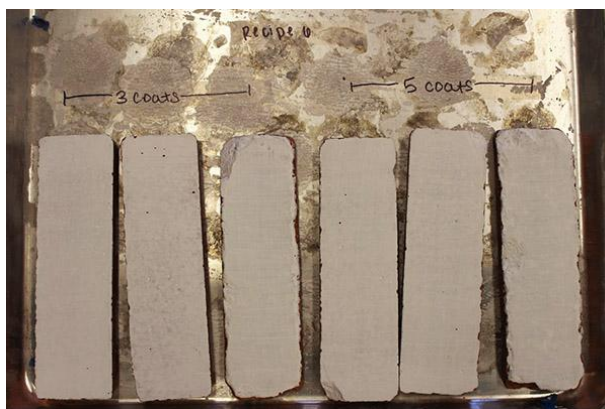


Figure 19. Measuring and adding the alum to limewash to make Recipe 6

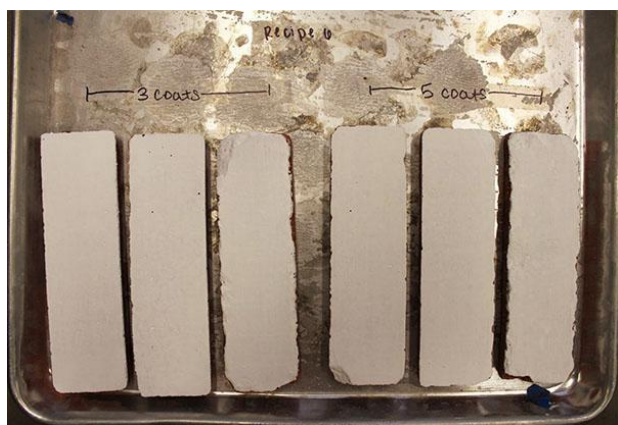
Recipe 6 was prepared in largely the same manner as the control limewash. Relatively the same amount of water (229.98g) and lime (118.76g) were mixed. This yielded 310mL of limewash. It was necessary to know the volume of the limewash prepared, because Recipe 6 (and all other recipes found that called for the addition of alum) gave a weight of alum to add to a certain volume of limewash. Therefore, based on 310ml of limewash, 9.29g of alum were added. The mixture brushed onto the brick easily and evenly, but it appeared that some undissolved alum particles clung to the brush. This did not affect the brick samples, because the undissolved particles did not come off onto the brick. Samples coated with Recipe 6 had good hiding power even after only two coats as seen in the photos below. There was not much subsequent change in appearance between coats 2-5.



a.



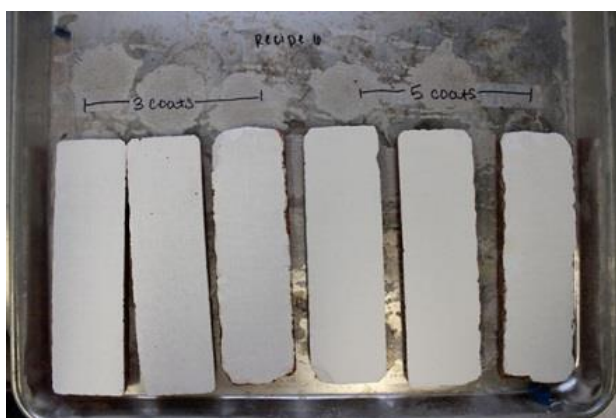
b.



c.



d.



e.

Figure 20. Recipe 5 after a) one, b) two, c) three, d) four, and e) five coats. Only the three bricks on the right of d. and e. received four and five coats. The three left brick have three coats each.



### 6.3 Specific Testing Procedure

ASTM D3359 and D4214 tests were performed three times each: three days, one week, and two weeks after the final coat. ASTM E96 was begun ten days after the final coat was applied. The full ASTM standards are reproduced in Appendix C. The samples were misted every 48 hours throughout testing in order to ensure full carbonation of the limewash. None were misted on the day of testing.

#### Chalking Resistance Test

To perform the chalking resistance test, Test Method D from ASTM D4214 was used. Transparent tape, black construction paper, and the limewash samples were gathered as materials. The test method consists in applying the transparent tape (2.5 to 4 inches long) to the limewash, smoothing it with uniform pressure from a finger, and then removing it. After this, the piece of tape was laid on the piece of black construction paper.<sup>86</sup> The tape was compared to Photographic Reference Standard No. 2 in the ASTM standard.

As suggested by the standard, if a sample appeared to be between grades, the intermediate number was used. The scale ranged from 0-8 with 0 being the greatest amount of chalking and 8 the least. The comparison results were all recorded in a spreadsheet to be analyzed at a later time. As mentioned previously, this test was performed on each brick sample three times: three days, one week, and two weeks after the final coat was applied.

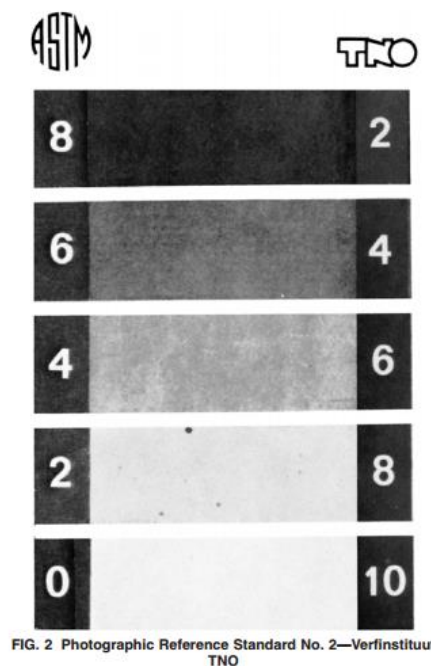


Figure 21. Photographic Reference Standard No. 2 from ASTM D4214

<sup>86</sup> ASTM D4214 calls for either black velvet or black construction paper. For the purposes of this test, construction paper was used so that the pieces of tape could be saved and used for later comparison.



Figure 22. Performing the chalking resistance test (ASTM 4214)

## Adhesion Test

For the adhesion test, Test Method B - Cross-Cut Tape Test was used. The ASTM standard is intended for metallic substrates, but it was adapted for this testing on brick. The materials gathered for ASTM 3359 were a scalpel, metal ruler straightedge, Semicro CHT tape, rubber eraser, hand lens, and limewash samples. The standard specifies Permacel 99 as the tape to be used for this test, but as that tape is no longer available, Semicro CHT tape was specifically designed by M.E. Taylor Engineering, Inc. to be a suitable replacement.<sup>87</sup>

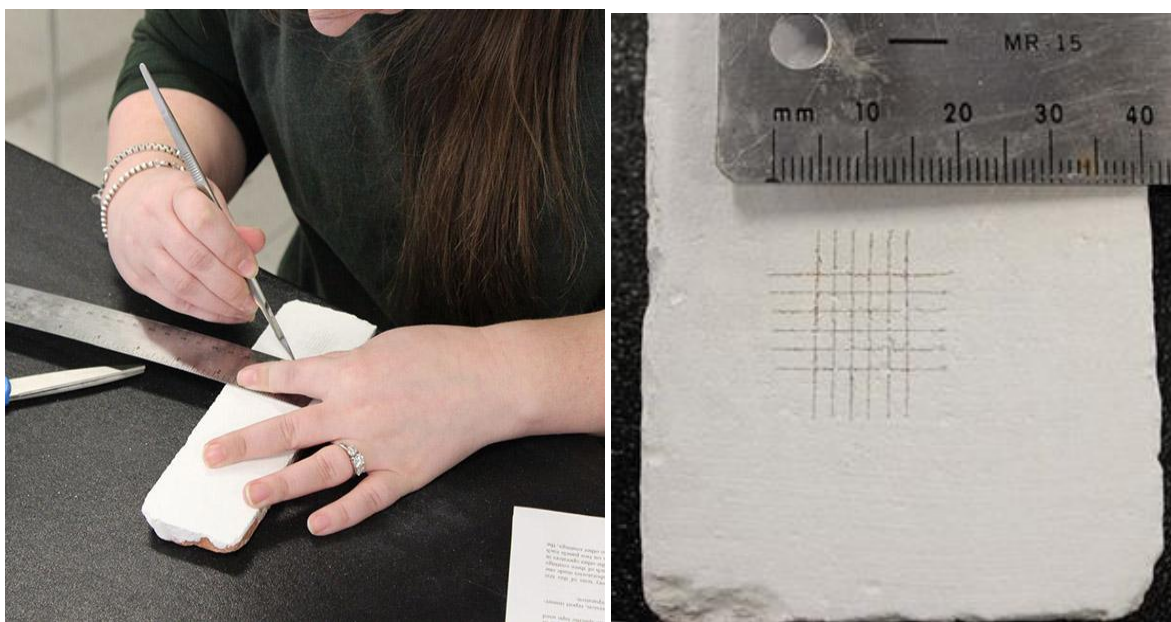


Figure 23. Cutting the cross-hatching lines to perform the adhesion test (ASTM 3359)

To carry out the test, six parallel lines, 2mm apart and 20mm long, were cut with the scalpel into the limewash until the brick substrate was reached. Six identical lines were made perpendicular to the first set, creating a cross-hatch. Any flakes were swept away with a soft brush, then a piece of Semicro CHT tape approximately 3 inches long was laid directly over the cross-hatched area. The eraser on a pencil was used to rub the tape to ensure proper contact with

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<sup>87</sup> Hester, Robert. 2011. 'Teaching an Old Standard New Tricks', *Adhesives & Sealants Industry Magazine*, accessed March 2, 2015 <<http://www.adhesivesmag.com/articles/90136-teaching-an-old-standard-new-tricks>>.

the limewash. After the tape had been on the substrate for 90 seconds, it was removed at almost a 180° angle. The cross-hatched area was then examined and compared to Classification of Adhesion Test Results (Figure 25).



Figure 24. Smoothing the Semicro CHT tape with an eraser and then removing tape

ASTM gives both a diagram and wording to help classify which category a sample should fall within. The scale ranges from 0B to 5B with 0B as the least adhesion and 5B as the most. These results were recorded in a spreadsheet along with other relevant facts such as where the failure occurred, patterns noted, or any anomalies. As with the chalking resistance test, ASTM 3359 was performed on the same samples three times: three days, one week, and two weeks after the final coat of limewash was applied.

CLASSIFICATION OF ADHESION TEST RESULTS		
CLASSIFICATION	PERCENT AREA REMOVED	SURFACE OF CROSS-CUT AREA FROM WHICH FLAKING HAS OCCURRED FOR SIX PARALLEL CUTS AND ADHESION RANGE BY PERCENT
5B	0% None	
4B	Less than 5%	
3B	5 – 15%	
2B	15 – 35%	
1B	35 – 65%	
0B	Greater than 65%	

FIG. 1 Classification of Adhesion Test Results

Figure 25. Classification of Adhesion Test Results in ASTM D3359



## Water-Vapor Transmission Test



Figure 26. Water-vapor transmission dish assembly

The final test, ASTM E96 Water-Vapor Transmission, was begun ten days after the final coat was applied to each core sample. Samples were misted every 48 hours to ensure full curing. The dish assembly was carried out after helpful instructions were given by Jennifer Schork, Conservation Associate with Integrated Conservation Resources, Inc. who has significant experience preparing samples for this test. Since ASTM E96 requires a large amount of samples and preparation, only Recipes 1-3 were tested, each in three and five coats. Brick

thins without any limewash applied were also tested in order to obtain baseline water-vapor transmission.

The setup for ASTM E96 requires the preparation of a dish consisting of a sample placed above a sealed container of water. This container can contain either a desiccant or water in order to monitor the rate at which vapor passes through the sample on top. For the purposes of this thesis, the water method was used. As mentioned in Section 6.1, 45mm cores of the brick thins were taken with a drill then painted with limewash. In order to match these 45mm cores, 50ml tri-corner beakers with a 47mm diameter opening were chosen as the dish. Other materials required for the water-vapor transmission test were Teflon tape and hot glue.

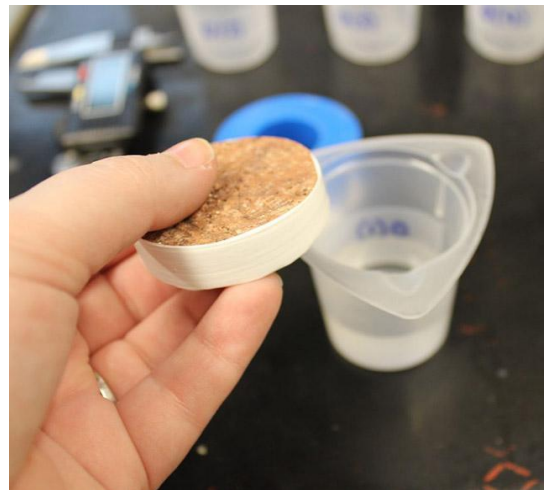


Figure 27. Brick core wrapped with Teflon tape





Figure 28. Hot gluing the brick core to the tri-corner beaker

To prepare the samples, modified ASTM E96 standard procedures were followed. Each tri-corner beaker was filled with 20mL of water. The brick samples were each wrapped three times around with Teflon tape; care was taken not to overlap the tape on either face of the sample. The core sample was then snugly placed on the lip of the tri-corner beaker. In order to fully create a seal, hot glue was placed around the rim of the dish assembly with the glue touching the Teflon tape and plastic beaker all the way around. Weight, temperature, and relative humidity measurements were taken immediately. The samples were all kept in a small room with stable relative humidity. Afterwards, the samples were not weighed for four days in order to adjust to the dish assembly.<sup>88</sup> Then, weight and relative humidity measurements of each dish assembly were taken beginning at 2:30pm each day for 2 weeks. After these measurements were completed, the water-vapor transmission rate was calculated according the ASTM E96 formula.

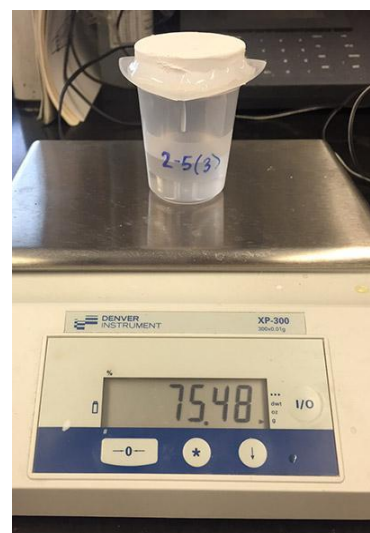


Figure 29. Weighing a dish assembly

<sup>88</sup> This was at the suggestion of Jennifer Schork, Integrated Conservation Resources.

## 7. TEST RESULTS

The full data and raw results from ASTM tests D3359, D4214, and E96 are listed in Appendix B. From a quantitative standpoint, some relevant information about the testing results is immediately apparent. As discussed in Chapter 5.3, for ASTM standards D3359 and D4214, half of the Old Mill Brick Thins used were rough and more porous (closer to historic brick properties) and the other half had a smoother and harder finish (modern brick properties). This did not cause any difference in the chalking resistance test. However, it had great ramifications for the adhesion test. Because of this difference in adhesion performance, it was necessary to separate the rough and smooth bricks for adhesion analysis. Since there was not a difference in chalking resistance results between the rough and smooth samples, there is no need for separate analysis.

### ASTM D4214

The chalking resistance results, based on the ASTM Reference Chart No. 2 (Figure 30), can first be broken down into the average data for the recipes (the average of three tests each for 3 coats and 5 coats).

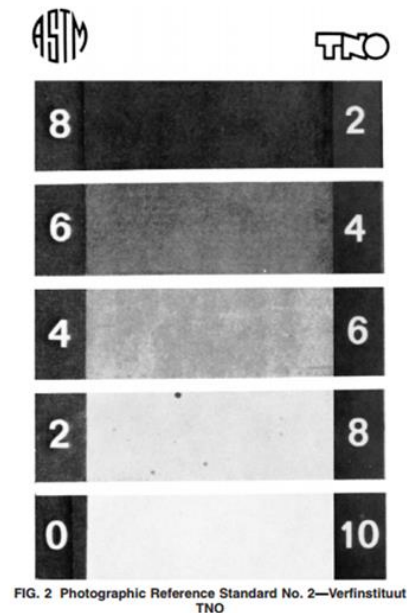


Figure 30. Photographic Reference Standard No. 2 from ASTM D4214

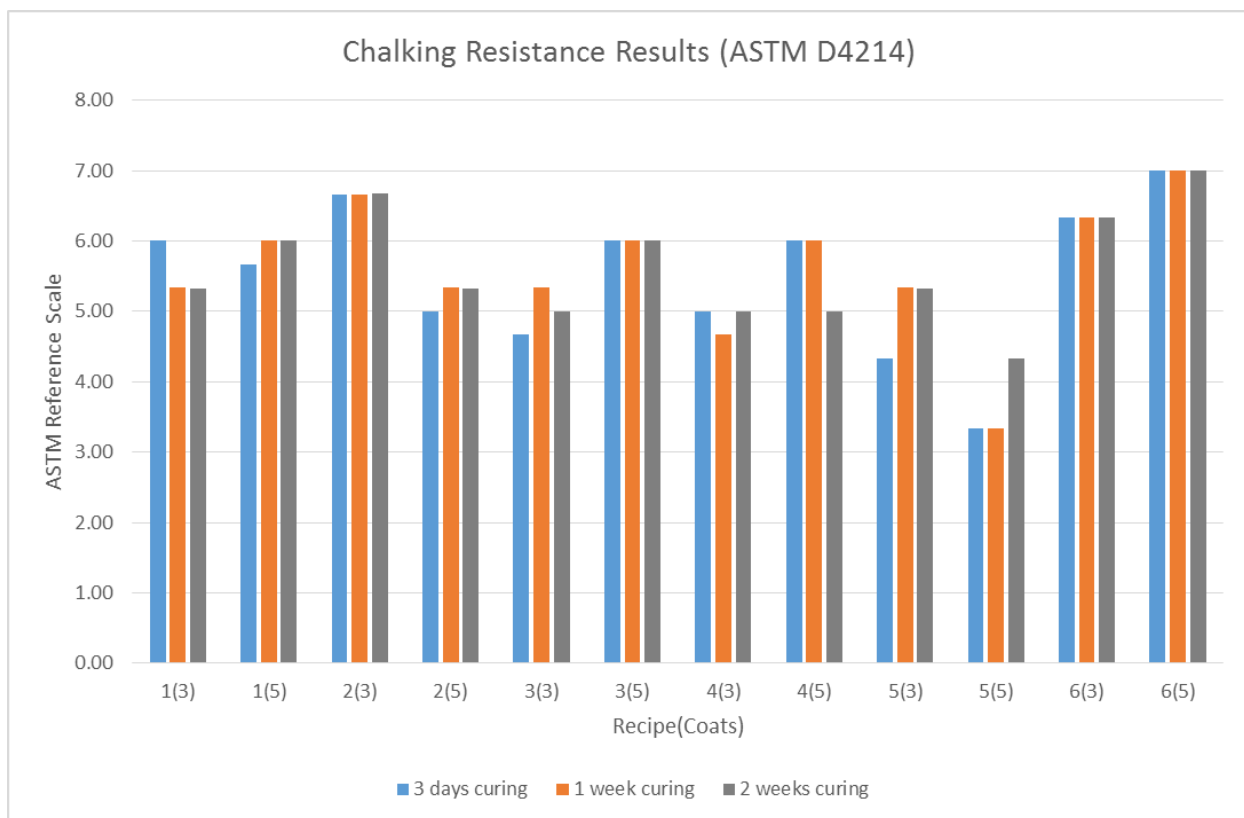


Figure 31. Bar graph of chalking resistance results – The average was taken of the triplicate samples to obtain these results for 3 days, 1 week, and 2 weeks after the final coat was applied.

As a reminder, the ASTM reference chart is scale of 0-8 with 0 being the largest amount of chalking and 8 the least. No samples were graded on the extreme ends of the spectrum as either a 0 or 8. Figure 31 shows that while there are small fluctuations in the amount of chalking based on curing time, generally the chalking rating was consistent. For the most part, chalking resistance either remained the same or within 1 point on the reference chart.

Based on these results, some recipes performed better overall than others. Recipe 6 was a clear standout that resisted chalking very well. After both three and five coats, it showed very little chalking. Recipe 2 with three coats also performed well, but after five coats the same recipe only showed fair results. This does not seem to be a trend amongst the washes though. Some washes performed better after five coats, but some performed worse. Recipe 5 displayed the most

chalking of all recipes, especially after five coats.<sup>89</sup> It is also useful to look at a graph of the complete data.

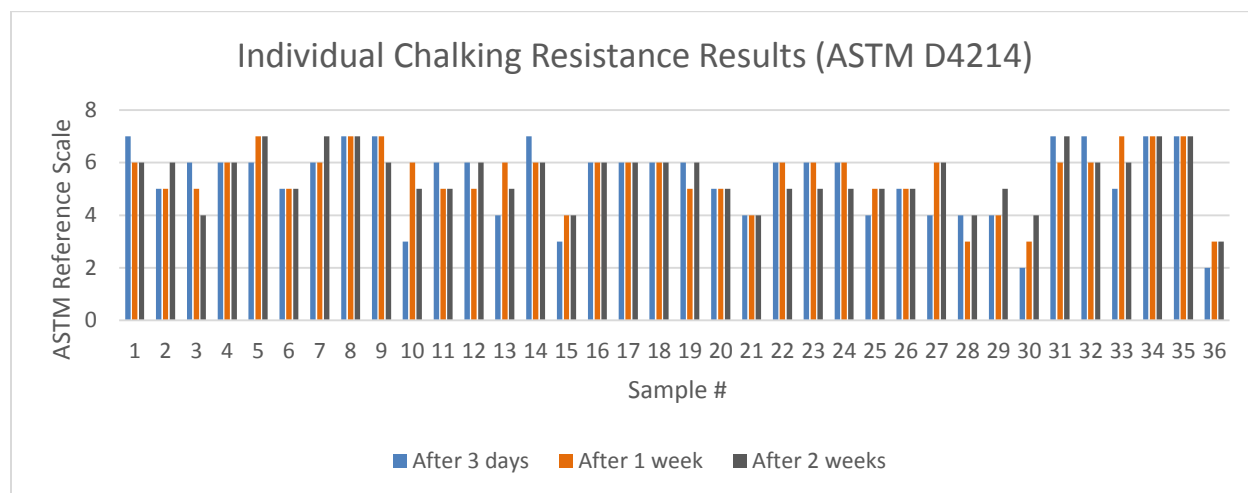


Figure 32. Bar graph of the individual chalking resistance results for each sample 3 days, 1 week, and 2 weeks after the final coat was applied

This chart shows that the control limewash, Recipe 1, performed moderately well under all circumstances. The other test recipes did not consistently perform either better or worse than the control; the results were mixed. While some of the chalking resistance results were erratic, it is generally possible to determine how well the other recipes performed in relation to the control limewash. Recipes 2 and 3 seem to have better chalking resistance than Recipe 1, while Recipe 5 performed worse. Recipe 4's results were about the same as the control wash.

### ASTM D3359

As mentioned, it was necessary to look at the adhesion test results not only by recipe and curing time but also based on whether the brick samples were rough or smooth. The two bar graphs below show a clear difference between rough and smooth surfaced bricks.

<sup>89</sup> This fact is reinforced by the adhesion resistance test as well. While Recipe 5 performed well in adhesion to the brick substrate, the top layers of paint were always pulled off with the tape.

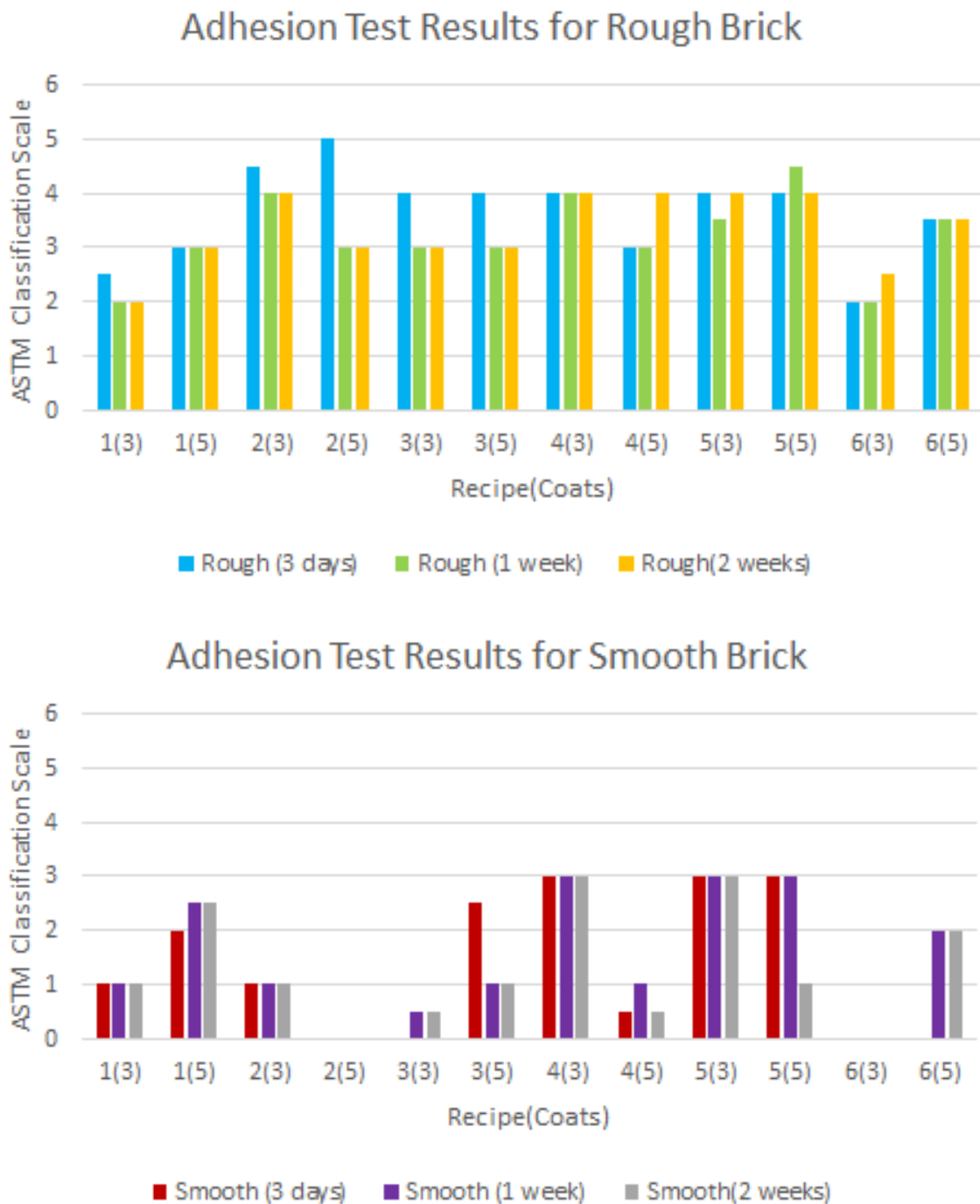


Figure 33. Two bar graphs depicting the average adhesion test results for rough brick and smooth brick separately – These graphs show that the adhesion results for smooth brick was significantly lower than rough brick.

Limewash had much better adhesion to the rough brick than to the smooth brick. This is not a surprising finding, because limewash performs best when it has a porous surface with which to interact. However, it is surprising *how* poorly the limewash adhered to the smooth brick. The adhesion of the washes to the smooth surface was so poor that simply cutting the cross-hatch lattice with a sharp scalpel, as specified in the ASTM standard, caused some of the limewash to chip off. For



Figure 34. Recipe 2 after five coats showing most of the lattice removed

most of the recipes, the tape further removed a great deal of coating. Some recipes performed so poorly that most all of the lattice was removed. Figure 34 shows one example of this. The only limewash that performed moderately well on the smooth brick was Recipe 5.

CLASSIFICATION OF ADHESION TEST RESULTS		
CLASSIFICATION	PERCENT AREA REMOVED	SURFACE OF CROSS-CUT AREA FROM WHICH FLAKING HAS OCCURRED FOR SIX PARALLEL CUTS AND ADHESION RANGE BY PERCENT
5B	0% None	
4B	Less than 5%	
3B	5 - 15%	
2B	15 - 35%	
1B	35 - 65%	
0B	Greater than 65%	

FIG. 1 Classification of Adhesion Test Results

Figure 35. Classification of Adhesion Test Results in ASTM D3359

By contrast, all of the limewashes had good adhesion to the rough bricks, with all ratings being between 3B and 5B. It is important to remember that the difference between category 5B and 4B is extremely small, and 3B can also indicate little removal of the coating. Of the samples on the more porous brick, only Recipe 1 after 3 coats performed as poorly as limewash on the smooth brick. As Recipe 1 is the control sample of only lime and water, this shows that all of the recipes improved adhesion for rough brick. The same was not true for smooth surfaced brick face however. This is potentially because those recipes were never meant or developed for modern brick.

It is useful to look at the adhesion test results by time period as well. There was some variation between 3 days and 1 week, although no clear trend. However, between 1 week and 2 weeks curing time, there was little variation at all. Only three samples displayed slightly different results from 1 to 2 weeks, but all the rest showed the same amount of adhesion. Limewashes, at least under laboratory conditions, should reach their full curing time by 1 week after the final coat. Otherwise, there would likely still be variability in the adhesion results after 2 weeks.

The ratings and charts only give information about how well the limewash adhered to the substrate, but the notes section for adhesion test results in Appendix B also gives some extra qualitative information. For example, Recipes 4 and 5 may have adhered to the substrate well, but the top and sometimes middle layers were often removed, leaving the bottom one or two layers behind. The same effect happened to Recipe 1, the control wash, but did not occur with Recipes 2 and 3. By contrast, the tape removed those washes down to the substrate.

## **ASTM E96**

The hypothesis going into water-vapor transmission was that the recipes with additives would have lower rates than pure limewash. This theory was echoed by Mold and Godbey in their 2005 paper stating, “Additives to limewash can affect the limewash porosity and permeability.”<sup>90</sup> Boxall and Trotman proved it to be true for limewashes containing linseed oil and tallow in their 1996 study.<sup>91</sup> However, they did not look at the effects of additives such as salt, zinc sulfate, ground rice, and the other combinations of ingredients found in Recipes 2 and 3, nor the effects of multiple coats. The water-vapor transmission tests completed for this thesis were able to further confirm that additives do have an effect on a limewash’s permeability.

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<sup>90</sup> Mold and Godbey, 8.

<sup>91</sup> Boxall, J. and Trotman, P.M. 1996. Limewash: Water Vapour Permeability Testing, 'JOCCA: Surface Coatings International', **79**(3): 128-129.

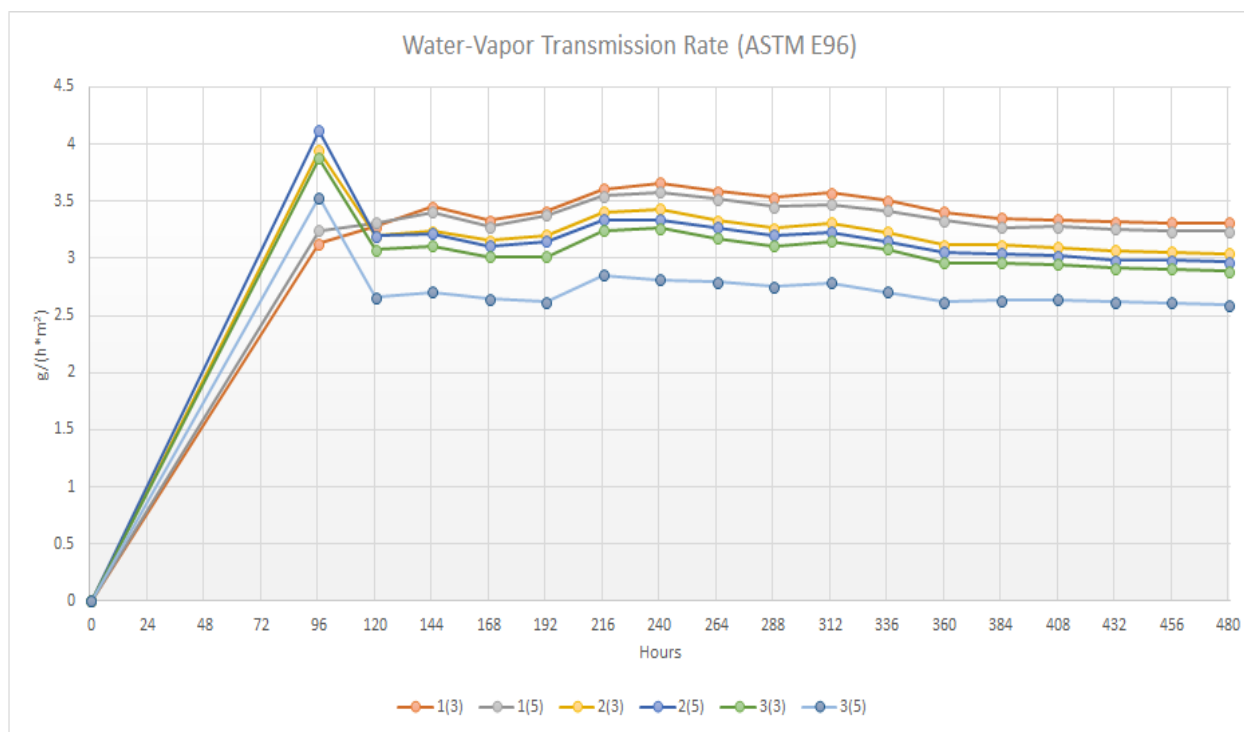


Figure 36. Graph of the water-vapor transmission rate for Recipes 1-3 - To obtain these numbers, the samples were weighed every 24 hours and the weight was put into the formula specified in ASTM E96 to obtain water-vapor transmission rate.  $WVT = \text{weight change} / (\text{time in hours} \times \text{cup mouth area in m}^2)$

This chart shows the order of water-vapor transmission rate from highest to lowest as Recipe 1, 2, and 3. For each recipe, the samples with three coats had a higher rate than five coats. Samples of uncoated rough brick were also tested, but the results seemed incongruous and were not included on the graph. The measurements for uncoated brick are listed in Appendix B. Uncoated brick was shown to have a lower water-vapor transmission rate than samples coated with Recipe 1. The reason for this difference might be that while smooth surfaced brick was used for all coated samples, rough brick was used for the untreated samples. This was only done out of necessity as there were no more smooth surfaced bricks available. Therefore, unfortunately it was not possible to directly compare the coated samples to uncoated rough brick transmission rates and the rate was not included on this graph.

Except for this uncoated brick, all of the samples behaved in the anticipated manner. The control limewash had the highest rate, followed closely by Recipe 2 with salt, zinc sulfate, and



milk. The additives in Recipe 2 were not expected to affect the water-vapor transmission rate as much as the thicker additives such as ground rice paste and hide glue in Recipe 3. This hypothesis was proved true. The rates for each sample were all close in value except for Recipe 3 with five coats as seen in the graph. The following chart shows the water-vapor transmission rate for each sample.

<b>Sample</b>	<b>Water-Vapor Transmission Rate in g/(h*m<sup>2</sup>)</b>
1(3)	3.31
1(5)	3.24
2(3)	3.04
2(5)	2.97
3(3)	2.89
3(5)	2.59

These test results prove that additives, as well as multiple coats, reduce the water-vapor transmission of a limewash. As historic structures were frequently limewashed on a yearly basis, the buildup of layers, and potentially different recipes, could have significantly impeded the breathability.

## 8. CONCLUSION

### 8.1 Discussion

It is possible to analyze Recipes 1-6 and compare the test results to historic claims. Some claims seem to be supported, but others do not. It is useful to acknowledge qualitative information about the recipes as well such as workability, aesthetics, and potential side effects of different additives.

#### **Recipe 1 - Control wash**

As the control wash, Recipe 1 establishes a baseline by which to compare the other recipes. As previously established, plain limewash without any additives would have been common in the 19th century. It is useful to know how it would have performed on brick surfaces, but it is also important to keep in mind that the performance of limewash (pure limewash or any of the modified limewashes) would have also been different on wood substrates. While this thesis does not examine those effects, this is a potential area of study for the future. The three iterations of the tests performed on Recipe 1 show that it performs moderately well in chalking resistance and fair to poor in adhesion on the ASTM scales for those tests. Additionally, it has a high water-vapor transmission rate as is expected of a limewash. The water-vapor transmission tests establish that the rate for 5 coats is less than 3 coats. While this is intuitive information, it had not been tested in a laboratory. This information sets a baseline that allows the other modified limewashes to be compared to the control wash.

#### **Recipe 2 – “Good for outdoor exposure” and “harden and prevent cracking”<sup>92</sup>**

The historic claims for Recipe 2’s benefits are vague; “good for outdoor exposure” and “harden[ing]” are not clearly testable assertions. However, it is safe to assume that a surface

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<sup>92</sup> Burn, Robert Scott, (ed.) 1871. *The New Guide to Masonry, Bricklaying and Plastering Theoretical and Practical*, London: Fullerton, 407.

coating good for outdoor work would have good adhesion as well as water-vapor transmission. Additionally, the additives causing the coating to “harden” could be interpreted to mean that the surface was less likely to chalk. Basically, the historic sources claim that Recipe 2 should be an improvement over pure limewash in at least chalking resistance and adhesion and on par with its water-vapor transmission. Based on the test results, this was true for chalking resistance and for adhesion to the rough brick. Recipe 2 was generally the best mixture for chalking resistance and one of the best for adhesion to rough brick. It performed very poorly for adhesion to modern brick, however. If this recipe were to be used in today’s world, it would not be recommended for modern, smooth brick use due to its serious lack of adhesion to the substrate. It could perform quite well on a more porous brick or historic brick though. Of course, more testing would need to be completed to understand how Recipe 2 would weather and affect the substrate, but the initial results for chalking resistance and adhesion are promising. In terms of water-vapor transmission, this mixture had a slightly lower rate than the pure limewash but not by a great amount. The difference between Recipe 2 and Recipe 1 with the same amount of coats is  $0.27 \text{ g}/(\text{h}\cdot\text{m}^2)$ . While this is a fairly small difference, it is clear that the addition of salt, zinc sulfate, and milk do affect water-vapor transmission rate. Based on the results, more coats would further reduce the rate. One other important characteristic to note about Recipe 2 is that it is more of a cream color while the control wash is pure white.

### **Recipe 3 - “Whitewash used at the White House”<sup>93</sup>**

Historic sources do not make any claims about the performance of Recipe 3. They only state that it was the limewash recipe used on the White House, with one source stating that this was the formula used by the United States government on other structures such as lighthouses.<sup>94</sup>

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<sup>93</sup> John Agg Papers, 1830.

<sup>94</sup> Hodgson, 57.

While it is difficult to actually trace the origins to the White House,<sup>95</sup> this recipe was reproduced in newspapers and journals many times throughout the 19th century. Interestingly, none of the sources ever comment on the performance of the limewash. Presumably, the White House pedigree was enough to vouch for its benefits. One source from 1923 references the use of additives similar to the ones in Recipe 3 by stating, “I have never used or advised the use of glue or rice flour paste in whitewash, though such things may produce a better effect on exterior walls by giving the lime coating a fuller and better surface.”<sup>96</sup> However, this too is quite vague and does little to inform about the actual effects of such additives.

Therefore, without any written documentation, test results from this thesis are the only way to understand how Recipe 3 might have functioned. Even before testing, simply mixing Recipe 3 yielded some interesting qualitative information. The process for mixing was complicated, and it was difficult to coordinate the timing of adding ingredients. It was not easy to achieve even and thorough coverage with Recipe 3, at least for the first few layers. However, layers 4 and 5 finally achieved a nice opaque coating. So, if Recipe 3 really were used on the White House (or any building concerned with having an even appearance), it was likely to have at least 4 or 5 coats of limewash.

In terms of performance, Recipe 3 was largely middle of the pack. The adhesion test results were slightly better than the control limewash and on par with the other recipes. Its only standout quality was chalking resistance after 5 coats; Recipe 3 consistently performed well in that category. As expected, Recipe 3’s water-vapor transmission rate was significantly lower than the control wash due to the ground rice paste and hide glue additives. This was especially

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<sup>95</sup> Some sources (such as *Dr. Chase’s Recipes* from 1866, 325) cite *National Intelligencer* as the origin of the recipe. However, the author was unable to locate the recipe in *National Intelligencer*.

<sup>96</sup> Kelly, Ashmun. 1923. *The Household Painter*, Philadelphia: David McKay Company, Publishers, 139-140.

true after 5 coats. The difference between Recipe 3 and 1 with three coats was 0.42 g/(h\*m<sup>2</sup>). With five coats, the difference was 0.65 g/(h\*m<sup>2</sup>). A buildup of coats over a number of years could seriously impede the breathability of a wall coated with Recipe 3. Based on these facts, coupled with the difficulty of mixing this modified limewash, it would not be a highly recommended recipe to use today. Additionally, like Recipe 2, this mixture is closer to a cream color than pure white, which may or may not be desired, depending on the intended application.

#### **Recipe 4 - “Harden the wash and thus improve its wearing qualities”<sup>97</sup>**

Salt, the only additive in Recipe 4, was by far the most common ingredient mentioned in the recipes collected for this thesis. It was both combined with other ingredients and used alone with lime and water. For mixtures with only salt, such as Recipe 4, historic sources state the benefits in different ways: “very permanent, does not crack, nor come off upon one’s hands or clothes,”<sup>98</sup> “salt hardens the lime,”<sup>99</sup> “adhere to the walls and destroy insects,”<sup>100</sup> and “harden the wash and thus improve its wearing qualities.”<sup>101</sup> Basically, these statements indicate that salt should prevent chalking by “hardening” the surface as well as aid its adhesion.

Despite these claims, the salt only slightly increased chalking resistance compared to the control limewash. The discrepancy may lie in the substrate used. It is possible that the historic claims for salt in limewash may have been intended for wood rather than brick. This is supported by the source cited above that claimed salt makes limewash “very permanent, does not crack, nor come off upon one’s hands or clothes,” because it goes on to say “The experiment was made

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<sup>97</sup> Ibid., 139.

<sup>98</sup> ‘Durable Whitewash’, *New England Farmer and Gardener’s Journal*, **XIII**(30): 235, 1835.

<sup>99</sup> Dr. Geo. Blakelee. *Blakelee’s Industrial Encyclopedia*, New York: Universal Information Exchange, Publishers, 1887, 162-167.

<sup>100</sup> *Morning Chronicle*, 1803.

<sup>101</sup> Kelly, 139.

only on wood.”<sup>102</sup> Therefore, while salt did increase the chalking resistance of limewash on brick by a small amount, the effect may be greater on wood. This would be an area of further study to pursue.

Salt had a greater effect on the adhesion of limewash. It performed a great deal better than the control limewash as well as better than most other recipes, along with Recipe 5. Salt did not change the appearance of the limewash as its aesthetic qualities were roughly the same as the control limewash. However, when using this recipe on brick in a real world environment, it could potentially lead to structural problems due to salts during freeze-thaw cycles. Weathering tests, as well as observations in real world applications, would need to be done before Recipe 4 could be recommended for brick substrates. Its good adhesion performance and bright white color does make this a promising formula though.

#### **Recipe 5 – “Adhere splendidly”<sup>103</sup>**

Limewash recipes containing sugar or molasses state that it will render lime more soluble, causing it to penetrate the substrate more<sup>104</sup> and thus adhere to the surface better.<sup>105</sup> The ASTM 3359 test results show this to be a valid claim. Recipe 5 performed the best overall in adhesion and even performed acceptably on modern brick, where the other recipes did not. This modified limewash certainly lived up to the historic claims.

However, it is unsurprising that the sources did not mention anything about the chalking resistance of a limewash with sugar added. Recipe 5 performed the worst by far in the ASTM 4214 tests. This was especially true after 5 coats; no other limewash recipe came close to chalking as much as this one. Interestingly though, while the top layers were easily removed with

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<sup>102</sup> ‘Durable Whitewash’, 235.

<sup>103</sup> ‘Sugar and Mortar’, 57.

<sup>104</sup> Walker, 142-156.

<sup>105</sup> ‘Sugar and Mortar’, 57.

tape, the bottom layer remained strongly adhered to the substrate. Therefore, Recipe 5 would not be recommended for use with numerous layers. As the testing was not performed using only one or two coats, it is impossible to know if it would perform better in chalking resistance tests under different conditions. The good adhesion properties of Recipe 5 are promising enough to look into its performance using one or two coats. The only potential drawback is that, as seen in Figures 18a and 18b, one to two coats of Recipe 5 do not provide excellent hiding power. However, a more transparent coating of limewash may be desired for certain applications.

**Recipe 6 – “Prevents it rubbing off”<sup>106</sup> and “give binding and hardening properties”<sup>107</sup>**

By far, most of the claims about limewash modified with alum state that it will prevent limewash from rubbing off. Only one source, the National Painters Magazine in 1908, claims that alum will also “give binding and hardening properties.” The reason for this is demonstrated by the testing. Recipe 6 performed the best of all limewashes in the chalking resistance test, but it did not perform particularly well in the adhesion testing. Its performance on the rough brick was fair, while it received some of the lowest rating for modern brick adhesion. Another claim made about the addition of alum was that it would render thatch incombustible,<sup>108</sup> while this claim was not tested for this thesis, it would be an interesting line of inquiry to pursue.

In terms of aesthetics, Recipe 6 was on par with pure limewash, which had the excellent hiding power. After only two coats, the brick substrate was not visible, and subsequent coats served to make the white color even brighter. Therefore, this recipe could be used for anyone desiring an opaque, bright white finish that will not rub off brick. Due to the lack of adhesion to the modern brick, this would only be recommended for historic or more porous modern brick.

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<sup>106</sup> Walker, 142-156.

<sup>107</sup> National Painters Magazine. 1908. *1000 More Paint Questions Answered*. New York: The Painters Magazine.

<sup>108</sup> ‘Incombustible Thatch’, *The Journal of the Franklin Institute*, **25**: 348-349, 1838.

## 8.2 Recommendations for further research

As intimated in the discussion of each recipe, there are many useful areas of further research that could be pursued. One of the most obvious courses of pursuit would be to observe the effects of weathering samples of each recipe. NCPTT performed QUV weathering to understand the durability of different modified limewashes; however, these were not the same recipes as tested for this thesis.<sup>109</sup> Therefore, performing QUV weathering on Recipes 1-6 would be a good way to compare these recipes to other known research. In addition to QUV weathering, samples could be naturally weathered and then examined for both durability of the limewash and condition of the substrate.<sup>110</sup> This way one of the recipes could be fully recommended for use today once the effect on the substrate was known.

Along this vein, an interesting claim (both historic and modern) about limewash is that it is able to help consolidate deteriorating stone.<sup>111,112</sup> If this were true, there could be many new uses and places for limewash and/or modified limewashes. A breathable, antiseptic, non-permanent surface coating could be a desirable tool for conservators and preservationists, especially since it was historically common. However, there is not published research on this theory. Experiments, such as the ones listed in ASTM E2167 for Selection and Use of Stone Consolidants, could be performed to test properties of stone after the application of a limewash. There are also many ways to vary this line of research to fit different parties' needs.

Another direction for research is to carry out the testing plan from the thesis on wood substrates. NCPTT's 2005 study did use wood in addition to brick for the test samples, but again,

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<sup>109</sup> Jackson, 2005.

<sup>110</sup> Limewash samples from the author's 2014 summer internship at Governors Island National Monument are undergoing a natural weathering process and have been in situ since late July 2014. The samples are monitored periodically.

<sup>111</sup> Mold and Godbey, 2.

<sup>112</sup> Brown, W.M. 1909. 'How to Preserve Stonework', *Stone: An Illustrated Magazine*, **XXIX**: 13-15.



these were different recipes. They found that none of the limewashes were as durable on wood as on brick. It would be interesting to see if any of Recipes 1-6 were able to increase performance on wood, especially since it is hypothesized that Recipe 4 may have been more specifically meant for wood. If Recipes 1-6 were applied to wood, it would be useful to perform adhesion, chalking resistance, as well as weathering tests so that the results could be compared to both this thesis as well as NCPTT's study. Testing the limewash recipes on wood could be particularly useful for any practitioners, conservators, or even homeowners who may want to utilize limewash on a wooden structure, whether that is because limewash was the historic coating or they are specifically looking for the beneficial properties that limewash can provide.

Besides the laboratory testing of limewashes to learn how they may best be applied today, there are a number of historical research avenues that could be pursued. This thesis exhausted most online databases for sources and discussions of limewash in American publications, particularly in the 19th century. However, analyzing other sources such as surviving personal diaries or correspondence archives from the 16th-18th centuries could help to provide a better understanding of early limewash use in this country. Unfortunately, this is a rather monumental task, to scour existing archives in hopes that there may be a useful mention of limewash. Instead, this task may need to wait until a future time when more private and personal collections are digitized. Beyond American uses of limewash, synthesizing world literature would also be an enormous but extremely useful endeavor.

Another daunting yet likely rewarding research task would be to collect paint samples from historic buildings and analyze any limewashes found through cross-section microscopy to determine the composition. As mentioned in Section 3.1, paint analyses of historic buildings *will* often do this, but there are not enough publically available studies to garner much information.

Taking more samples, potentially in combination with gaining access to existing paint studies, would give concrete evidence of limewash composition. This data could be aggregated and analyzed according to region, time period, climate, and potentially even socio-economic status to reveal trends in additive use. Of course, in addition to time, effort, and cost, one of the major limitations of such a study is that the researcher is limited to existing buildings, which may or may not be an accurate representation of all historical limewash use.

Clearly, there are many further courses of study and research that could be pursued regarding limewash. Any one of the aforementioned projects may yield useful information about either the history of limewash, potential applications, or both. This thesis is only a start to understanding how limewash was used and functioned historically. However, limewash need not stay in the past. Water-vapor transmission tests from this thesis have shown that even with additives and multiple layers, limewash is a still highly breathable coating. The adhesion and chalking resistance tests indicate that certain additives truly can improve the performance of a limewash (albeit not all additives). Because of this information, combined with the ubiquity with which limewash was once used, preservationists and conservators may want to consider using this coating for more projects. Recipes, such as the ones tested for this thesis, could be tailored to fit the specific needs of a restoration, preservation, or rehabilitation project. Before this can happen though, more research is needed to learn the long-term effects of limewash or limewash recipes on historic building materials.

## APPENDIX A. TESTED RECIPES

Recipes Used in Adhesion (ASTM D3359), Chalking Resistance (ASTM D 4214), and Water-Vapor Transmission (E96) Tests

### Recipe 1 - Control

Sample 6 is the control sample of pure lime and water. In “Limewash: Compatible Coverings for Masonry and Stucco” from the *International Building Lime Symposium* 2005, Mold and Godbey state that the percentage of lime solids in a wash should be 15-20% with the remaining 80-85% being water. For this experiment, 17% lime and 83% water was used.

- Mold, P. and Godbey, R. 2005. ‘Limewash: Compatible Coverings for Masonry and Stucco’. *International Building Lime Symposium*, National Lime Association, Orlando, Florida, 9-11 March 2005.

Ingredient	Amount Used
Niagara Lime Putty	118.92g
Water	230.34g

Lime putty contains 50% water and 50% lime. Therefore, the actual amount of lime in this mixture is 59.46g. This is the formula base formula that was used for all other recipes.

## Recipe 2

“For outside work: Take quicklime half a bushel; slake it, and add one pound of common salt, and half a pound of sulphate of zinc, and a gallon of sweet milk. Dissolve the salt and zinc before mixing; thin the whole to a proper consistence with water, and it is ready for use.”

- Frederick Thomas Hodgson, *Plaster and Plastering. Mortars and Cements, How to Make, and How to Use*, New York: The Industrial Publication Company, 1883.

Ingredient	Historic Recipe Amount	Scaled Amount Used
Lime putty	38 lbs.	119.15g
Water	Not specified	215.3g
Milk	1 lb.	13.48g
Salt	0.5 lb.	1.5g
Zinc Sulfate	~8.6 lbs.	0.78g

Lime putty contains 50% water and 50% lime. Therefore, the actual amount of lime in this mixture is 59.57g.

### Recipe 3

“Whitewash used at the White House: Receipt for Making Wite was such as used on the Presidents House. Take half a bushel of unslacked lime, and slake it with boiling hot water, covering it over during the process. Strain it and add a peck of salt dissolved in hot water; three pounds of ground rice boiled to a thin paste put in boiling hot; half a pound of powdered Spanish whiting, and a pound of clean glue dissolved in water; Mix and let it stand for several days. Then keep in a kettle on a portable furnace and put it on as hot as possible with a painters or whitewash brush.”

- John Agg Papers. 1830. David M. Rubenstein Rare Book & Manuscript Library, Duke University Library. Cited in Johansen, E.L.A. 1999. ‘Deterioration of Gneiss Due to Limewash at Eastern State Penitentiary’. Thesis. University of Pennsylvania.

Ingredient	Historic Recipe Amount	Scaled Amount Used
Lime putty	38 lbs.	125g
Water	Not specified	230.5g
Salt	17.5g	27.4g
Ground rice	3 lbs.	4.7g
Whiting	0.5 lbs.	0.78g
Glue	1 lb.	1.6g

Lime putty contains 50% water and 50% lime. Therefore, the actual amount of lime in this mixture is 62.5g. More lime putty was added to compensate for the extra water added to dissolve the hide glue, salt, and ground rice.

## Recipes Used in Adhesion (ASTM D3359) and Chalking Resistance (ASTM D 4214) Tests Only

### Sample 4

“The formula for lump lime whitewash was about 7 pounds of salt to ½ bushel of lump lime  
The coarse salt was used.”

- Kelly, Ashmun. 1923. *The Household Painter*, Philadelphia: David McKay Company, Publishers.

Ingredient	Historic Recipe Amount	Scaled Amount Used
Lime putty	38 lbs.	119.35g
Water	Not specified	226.99g
Salt	7 lbs.	10.99g

Lime putty contains 50% water and 50% lime. Therefore, the actual amount of lime in this mixture is 59.68g.

## Sample 5

“For lime washing, one pound of sugar to sixteen gallons of water will make it adhere splendidly. Drops of whitewash made with water so mixed, if they fall on the floor or window, or an iron plate, cannot be washed off.”

- ‘Sugar and Mortar’. The Manufacturer and Builder, 22(3): 57, 1890

Ingredient	Historic Recipe Amount	Scaled Amount Used
Lime putty	16 gallons limewash	119.2g
Water		229.5g
Sugar	1 lb.	2.41g

Lime putty contains 50% water and 50% lime. Therefore, the actual amount of lime in this mixture is 59.6g. For this recipe, part of the historic measurement was by volume and part by weight. To reconcile this, the base limewash recipe was mixed then measured by volume (315ml). The amount of sugar to add was scaled based off of this volume measurement.

## Sample 6

“One-quarter pound of commercial alum is sufficient for one gallon of whitewash. ... The addition of alum is to give binding and hardening properties to the wash, especially on damp surfaces.”

- National Painters Magazine. 1908, *1000 More Paint Questions Answered*. New York: The Painters Magazine.

Ingredient	Historic Recipe Amount	Scaled Amount Used
Lime putty	1 gallon limewash	118.76g
Water		229.98g
Alum	0.25 lb.	9.28g

Lime putty contains 50% water and 50% lime. Therefore, the actual amount of lime in this mixture is 59.38g. For this recipe, part of the historic measurement was by volume and part by weight. To reconcile this, the base limewash recipe was mixed then measured by volume (310mL). The amount of alum to add was scaled based off of this volume measurement.



## APPENDIX B. RAW TEST DATA

### ASTM D4214 - Chalking Resistance

Sample#	Recipe-Coats(Triplicate#)	Test	3 days after final coat	1 week after final coat	2 weeks after final coat
1	1-3(1)	Chalking Resistance	7	6	6
2	1-3(2)	Chalking Resistance	5	5	6
3	1-3(3)	Chalking Resistance	6	5	4
4	1-5(1)	Chalking Resistance	6	6	6
5	1-5(2)	Chalking Resistance	6	7	7
6	1-5(3)	Chalking Resistance	5	5	5
7	2-3(1)	Chalking Resistance	6	6	7
8	2-3(2)	Chalking Resistance	7	7	7
9	2-3(3)	Chalking Resistance	7	7	6
10	2-5(1)	Chalking Resistance	3	6	5
11	2-5(2)	Chalking Resistance	6	5	5
12	2-5(3)	Chalking Resistance	6	5	6
13	3-3(1)	Chalking Resistance	4	6	5
14	3-3(2)	Chalking Resistance	7	6	6
15	3-3(3)	Chalking Resistance	3	4	4
16	3-5(1)	Chalking Resistance	6	6	6
17	3-5(2)	Chalking Resistance	6	6	6

18	3-5(3)	Chalking Resistance	6	6	6
19	4-3(1)	Chalking Resistance	6	5	6
20	4-3(2)	Chalking Resistance	5	5	5
21	4-3(3)	Chalking Resistance	4	4	4
22	4-5(1)	Chalking Resistance	6	6	5
23	4-5(2)	Chalking Resistance	6	6	5
24	4-5(3)	Chalking Resistance	6	6	5
25	5-3(1)	Chalking Resistance	4	5	5
26	5-3(2)	Chalking Resistance	5	5	5
27	5-3(3)	Chalking Resistance	4	6	6
28	5-5(1)	Chalking Resistance	4	3	4
29	5-5(2)	Chalking Resistance	4	4	5
30	5-5(3)	Chalking Resistance	2	3	4
31	6-3(1)	Chalking Resistance	7	6	7
32	6-3(2)	Chalking Resistance	7	6	6
33	6-3(3)	Chalking Resistance	5	7	6
34	6-5(1)	Chalking Resistance	7	7	7
35	6-5(2)	Chalking Resistance	7	7	7
36	6-5(3)	Chalking Resistance	2	3	3

## ASTM D3359 - Adhesion Tape Test

Sample #	Recipe-Coats(Triplicate#)	Test	After 3 days	After 1 week	After 2 weeks	Notes
1	1-3(1)	Adhesion	3B	3B	3B	Most of the failure was between the middle coats, not the base coat and substrate. More came off after 1 week than original.
2	1-3(2)	Adhesion	2B	1B	1B	Good amount of detachment at the substrate, some chipping of paint from the cutting but most removed with tape.
3	1-3(3)	Adhesion	1B	1B	1B	Chipping from just cutting, tape removed a lot of paint to the substrate.
4	1-5(1)	Adhesion	2B	3B	3B	Some chipping from cutting only, tape removed limewash to substrate as well as some top layers. Much less after 1 week than 3 days.
5	1-5(2)	Adhesion	3B	3B	3B	Mostly the tops layers came off with the tape but not much all the way to the substrate.
6	1-5(3)	Adhesion	2B	2B	2B	A lot of chipping to the substrate from cutting, tape removed a good amount as well.
7	2-3(1)	Adhesion	4B	4B	4B	Recipe 2 feels much more difficult to cut than Recipe 1.
8	2-3(2)	Adhesion	5B	4B	4B	Very little came off with either test
9	2-3(3)	Adhesion	1B	1B	1B	A lot of chipping from just cutting with the scalpel. Tape took off a lot of paint to the substrate.
10	2-5(1)	Adhesion	0B	0B	0B	Large chunks of limewash removed with just cutting, tape removed most all else.
11	2-5(2)	Adhesion	5B	3B	3B	Not much removed either after 3 days or 1 week..
12	2-5(3)	Adhesion	0B	0B	0B	Large chunks of limewash removed with just cutting, tape removed most all else.
13	3-3(1)	Adhesion	0B	1B	1B	Chips came off from cutting. After 3 days, the tape basically removed most of the lattice. After 1 week, not quite as much removed but still a good deal. Came off in small circles.
14	3-3(2)	Adhesion	4B	3B	3B	Not much removal for either
15	3-3(3)	Adhesion	0B	0B	0B	With both tests, the tape basically removed everything from the lattice.
16	3-5(1)	Adhesion	2B	1B	1B	Very little removed from cutting, but the tape seems to adhere better to this limewash than most and removed a lot of paint to substrate.
17	3-5(2)	Adhesion	4B	3B	3B	Very little came off

18	3-5(3)	Adhesion	3B	1B	1B	Much better adhesion after 3 days than 1 week.
19	4-3(1)	Adhesion	3B	3B	3B	A bit of chipping from cutting, but the taped did not remove much else.
20	4-3(2)	Adhesion	4B	4B	4B	Some detachment between middle layers, but very little removed with tape
21	4-3(3)	Adhesion	4B	4B	4B	Some detachment between middle layers, but very little removed with tape
22	4-5(1)	Adhesion	1B	1B	0B	Medium amount of chipping at most intersections of lattice from cutting only. A good deal removed with tape to the substrate.
23	4-5(2)	Adhesion	3B	3B	4B	This removal was mostly between the top and middle layers of limewash.
24	4-5(3)	Adhesion	0B	1B	1B	Less chipping from cutting after 1 week than after 3 days, but the tape removed a good deal from both. Better after 1 week.
25	5-3(1)	Adhesion	3B	3B	3B	A small amount of chipping from cutting. The tape only removed some of the top layer, but not down to the substrate.
26	5-3(2)	Adhesion	4B	3B	4B	After 3 days, small amount of chipping from cutting but the tape did not remove much else. After 1 week, the tape did not stick well to the limewash and didn't remove much.
27	5-3(3)	Adhesion	4B	4B	4B	Only the top layer came off, the lattice was largely intact.
28	5-5(1)	Adhesion	3B	3B	1B	Only a small amount of chipping, the tape mostly removed the top layer of limewash.
29	5-5(2)	Adhesion	4B	5B	4B	Very little removal all the way to substrate. After 3 days, most of the top layer came off. After 1 week, only a small amount of top layer removed.
30	5-5(3)	Adhesion	4B	4B	4B	Mostly just removal of top and middle layers of limewash.
31	6-3(1)	Adhesion	1B	2B	3B	A good amount of chipping, even with rough brick. Tape removed even more paint to the substrate.
32	6-3(2)	Adhesion	3B	2B	2B	Small bit of chipping. Most of the top layers came off and less all the way to substrate.
33	6-3(3)	Adhesion	0B	0B	0B	A great deal of chipping. Removal most of paint.
34	6-5(1)	Adhesion	3B	3B	3B	Top layer came off but not much all the way to the substrate.
35	6-5(2)	Adhesion	4B	4B	4B	Very little removed, some of top layer.
36	6-5(3)	Adhesion	0B	2B	2B	Medium to large amount of chipping, more removed after three days than 1 & 2 weeks

## ASTM E96 - Water-Vapor Transmission

Hours	0	96	120	144	168	192	216	240	264	288	312	336	360	384	408	432	456	480
Sample																		
0(1)	69.53	69.12	68.94	68.75	68.64	68.51	68.3	68.18	68.08	67.99	67.85	67.73	67.64	67.58	67.48	67.37	67.26	67.14
0(2)	70.26	69.93	69.76	69.58	69.47	69.34	69.15	69.02	68.89	68.8	68.65	68.56	68.49	68.39	68.28	68.17	68.06	67.94
0(3)	73.02	72.56	72.42	72.23	72.13	71.99	71.79	71.66	71.56	71.45	71.32	71.2	71.13	71.03	70.92	70.82	70.71	70.59
0(4)	71.69	71.24	71.08	70.89	70.79	70.63	70.42	70.29	70.18	70.07	69.92	69.82	69.75	69.66	69.53	69.41	69.3	69.18
0(5)	73.11	72.68	72.51	72.35	72.24	72.11	71.93	71.8	71.71	71.6	71.47	71.37	71.31	71.24	71.11	71.01	70.89	70.78
1-3(1)	75.88	75.37	75.22	75.05	74.94	74.78	74.59	74.42	74.29	74.18	74.03	73.92	73.84	73.74	73.62	73.5	73.37	73.24
1-3(2)	70.81	70.41	70.28	70.1	70.01	69.86	69.66	69.51	69.42	69.31	69.16	69.07	69	68.9	68.79	68.68	68.56	68.44
1-3(3)	77.45	77	76.86	76.71	76.62	76.47	76.28	76.12	76.03	75.93	75.78	75.69	75.62	75.51	75.42	75.31	75.19	75.07
1-3(4)	79.25	78.8	78.65	78.51	78.43	78.31	78.12	77.95	77.86	77.76	77.6	77.49	77.44	77.35	77.25	77.14	77.04	76.92
1-3(5)	73.3	72.72	72.55	72.37	72.24	72.06	71.85	71.7	71.56	71.42	71.26	71.15	71.05	70.95	70.79	70.67	70.52	70.38
1-5(1)	71.05	70.59	70.44	70.31	70.23	70.08	69.91	69.75	69.65	69.55	69.42	69.32	69.25	69.16	69.04	68.92	68.82	68.7
1-5(2)	72.16	71.69	71.55	71.41	71.33	71.17	70.99	70.86	70.76	70.65	70.52	70.42	70.35	70.26	70.14	70.04	69.94	69.82
1-5(3)	71.88	71.32	71.19	71.01	70.9	70.74	70.54	70.39	70.25	70.15	69.99	69.87	69.78	69.69	69.55	69.43	69.3	69.17
1-5(4)	75.81	75.32	75.19	75.05	74.94	74.78	74.59	74.43	74.33	74.23	74.08	73.98	73.89	73.8	73.66	73.56	73.45	73.32
1-5(5)	76.99	75.45	75.34	75.18	75.08	74.93	74.71	74.59	74.48	74.36	74.22	74.12	74.04	73.95	73.82	73.72	73.58	73.46
2-3(1)	72.33	71.79	71.68	71.52	71.4	71.23	71.04	70.9	70.81	70.69	70.53	70.44	70.38	70.25	70.13	70.01	69.89	69.78
2-3(2)	76.79	76.28	76.14	76	75.9	75.76	75.54	75.4	75.3	75.2	75.06	74.97	74.9	74.78	74.68	74.57	74.44	74.33
2-3(3)	74.42	73.99	73.86	73.75	73.67	73.55	73.37	73.24	73.16	73.08	72.94	72.86	72.8	72.7	72.6	72.52	72.42	72.32

2-3(4)	71.05	70.59	70.47	70.36	70.26	70.14	69.95	69.81	69.74	69.65	69.5	69.42	69.37	69.26	69.16	69.07	68.97	68.89
2-3(5)	73.86	73.38	73.25	73.11	73	72.88	72.7	72.55	72.45	72.35	72.21	72.12	72.07	71.95	71.84	71.74	71.64	71.53
2-5(1)	78.72	78.23	78.09	77.94	77.85	77.7	77.5	77.39	77.27	77.18	77.03	76.94	76.87	76.76	76.65	76.56	76.44	76.33
2-5(2)	72.72	72.26	72.14	72.03	71.94	71.82	71.64	71.52	71.42	71.33	71.2	71.12	71.06	70.95	70.86	70.77	70.66	70.56
2-5(3)	76.03	75.49	75.37	75.23	75.12	74.97	74.77	74.61	74.52	74.41	74.27	74.17	74.09	73.97	73.85	73.76	73.64	73.5
2-5(4)	75.85	75.31	75.2	75.08	74.98	74.86	74.69	74.55	74.45	74.36	74.22	74.15	74.1	73.98	73.89	73.8	73.68	73.59
2-5(5)	71.22	70.8	70.69	70.58	70.5	70.38	70.21	70.1	70.02	69.93	69.8	69.75	69.68	69.59	69.49	69.4	69.31	69.22
3-3(1)	73.02	72.5	72.29	72.14	72.04	71.92	71.7	71.59	71.4	71.42	71.26	71.16	71.12	71.02	70.92	70.82	70.71	70.61
3-3(2)	76.42	75.9	75.78	75.64	75.55	75.42	75.22	75.1	75	74.9	74.76	74.67	74.61	74.5	74.39	74.31	74.19	74.09
3-3(3)	77.05	76.56	76.48	76.36	76.27	76.15	75.95	75.82	75.74	75.65	75.52	75.44	75.38	75.26	75.16	75.06	74.96	74.86
3-3(4)	76.92	76.43	76.32	76.18	76.08	75.96	75.76	75.61	75.52	75.43	75.28	75.18	75.14	75.02	74.92	74.82	74.71	74.63
3-3(5)	70.7	70.29	70.16	70.06	69.97	69.88	69.7	69.58	69.5	69.41	69.28	69.21	69.17	69.07	68.97	68.89	68.79	68.7
3-5(1)	77.55	77.07	76.97	76.87	76.77	76.67	76.49	76.37	76.27	76.18	76.04	75.98	75.91	75.8	75.62	75.48	75.38	75.3
3-5(2)	72.14	71.73	71.64	71.53	71.45	71.36	71.18	71.07	71	70.91	70.79	70.73	70.68	70.58	70.49	70.42	70.32	70.24
3-5(3)	76.75	76.33	76.23	76.11	76.03	75.92	75.74	75.61	75.53	75.44	75.32	75.25	75.2	75.09	75	74.92	74.82	74.73
3-5(4)	76.72	76.39	76.32	76.22	76.15	76.08	75.92	75.8	75.74	75.66	75.55	75.5	75.46	75.36	75.28	75.22	75.14	75.06
3-5(5)	74.73	74.29	74.19	74.06	73.96	73.86	73.66	73.56	73.48	73.39	73.28	73.2	73.14	73.03	72.94	72.86	72.76	72.66

## **APPENDIX C. ASTM STANDARDS**

ASTM D4214-07 Standard Test Methods for Evaluating the Degree of Chalking of Exterior Paint Films

ASTM D3359-09 Standard Test Methods for Measuring Adhesion by Tape Test

ASTM E96/E96M-14 Standard Test Methods for Water Vapor Transmission of Materials



# Standard Test Methods for Evaluating the Degree of Chalking of Exterior Paint Films<sup>1</sup>

This standard is issued under the fixed designation D4214; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

## 1. Scope

1.1 These test methods cover the evaluation of the degree of chalking on white or tinted exterior paint films. These test methods describe the procedures recommended for transferring the chalk to a fabric or fingertip, which is then compared to photographic reference standards, or in the case of adhesive tapes, compared to a reflectance table or photographic reference standards, to determine the degree of chalking.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

**D662 Test Method for Evaluating Degree of Erosion of Exterior Paints**

**E1347 Test Method for Color and Color-Difference Measurement by Tristimulus Colorimetry**

### 2.2 Other Document:

**Pictorial Standards of Coating Defects<sup>3</sup>**

## 3. Terminology

### 3.1 Definitions:

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.25 on Evaluation of Weathering Effects.

Current edition approved July 1, 2007. Published July 2007. Originally approved in 1982. Last previous edition approved in 1998 as D4214 – 98 which was withdrawn March 2007 and reinstated in July 2007. DOI: 10.1520/D4214-07.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Copies of the pictorial photographic reference standards applicable to Test Method A are contained in the publication *Pictorial Standards of Coatings Defects* and may be obtained from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

3.1.1 *chalking, n*—the formation on a pigmented coating of a friable powder evolved from the film itself at or just beneath the surface.

## 4. Significance and Use

4.1 The procedures provide a broad range of techniques and photographic references to evaluate chalking of exterior paints.

## 5. Type of Chalking

5.1 Only one type of chalking is recognized, as defined in Section 3.

## 6. Use of Photographic Reference Standards

6.1 The photographic reference standards that are part of these test methods are representative of the degrees of chalking on a paint film. The photographs shown in Fig. 1 and Fig. 2 are for illustration purposes only and should not be used for evaluation.

6.2 The use of photographic reference standards illustrated in Fig. 1 and Fig. 2 requires the following precautions:

6.2.1 The degree of chalking will vary over any given area. Therefore, an average portion of the coating should be evaluated. On large surfaces, it is recommended that the rating be made at several locations and the mean and range reported.

6.2.2 It is difficult to make readings on a windy day and making readings at such time should be avoided. It should also be noted that rain, snow, or moisture in any form will remove chalk so that readings should be made after a period of clear weather and when the surface is dry.

6.2.3 Chalking and erosion (Note 1) are closely related. However, the rate of chalking as measured by these test methods, and the rate of erosion may not be comparable because some pigment combinations tend to retain chalk on the surface while other pigment combinations exert a self-cleaning action by natural means.

NOTE 1—For the evaluation of erosion, see Test Method D662.

6.3 Records may be kept on forms<sup>4</sup> such as shown in Fig. 3. Reporting of the results shall include the information given in Section 8.

<sup>4</sup> These record sheets may be obtained from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.



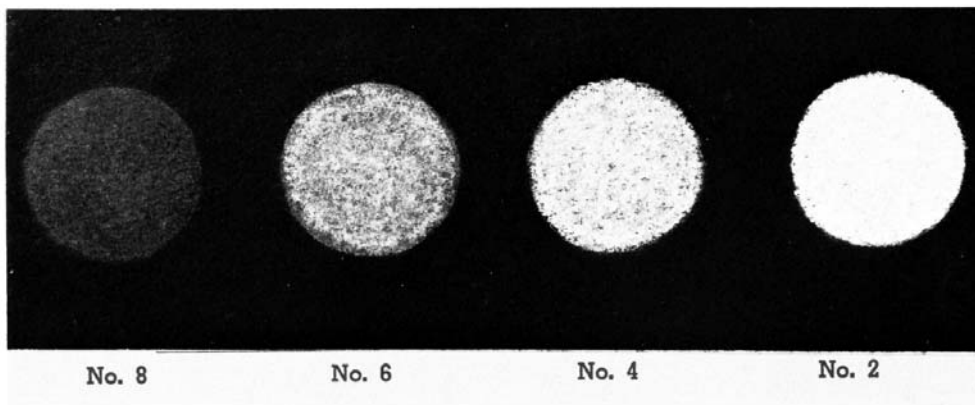


FIG. 1 Photographic Reference Standard No. 1—Test Method D659

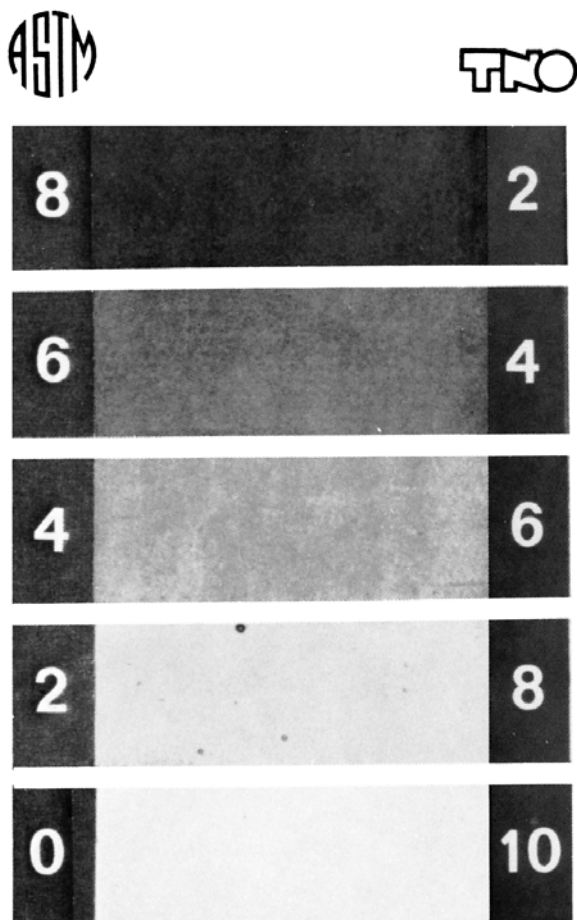


FIG. 2 Photographic Reference Standard No. 2—Verfijnstituut TNO

6.4 When these test methods is referenced in specifications for performance, the permissible degree of chalking is established between the producer and the user.

## 7. Recommended Procedures

### 7.1 Test Method A—Cloth Tape Method:

7.1.1 *Material*—Fabric, as agreed upon between the producer, user, or other interested parties, to rub against the

surface being tested. Black (or white for dark coatings) wool felt, velvet, and velveteen have proven particularly effective.

7.1.2 *Procedure*—Wrap the fabric around the index fingertip, then make a 50 to 75-mm (2 to 3-in.) stroke with medium pressure on the coating under observation. Remove the fabric and compare the spot of chalk on it with Photographic Reference Standard No. 1.

NOTE 2—Medium pressure can be quantified by placing the finger on a balance or scale, and pressing downward until 3 to 5-lb pressure is obtained.

### 7.2 Test Method B—Wet Finger Method:

7.2.1 *Procedure*—Moisten a fingertip and with medium pressure make one continuous rub 50 to 65 mm (2 to 2½ in.) in length on the surface under test. The chalk from this test method should be rated as None, Visible, or Severe; however, some may prefer to use an even numbered scale of 10 to zero.

### 7.3 Test Method C—Transparent Tape Method:<sup>5</sup>

#### 7.3.1 Materials:

7.3.1.1 *Cellulose Adhesive Tape*, 13-mm (½-in.) wide, pressure-sensitive.

7.3.1.2 *Eraser*, ¾ in. (20 mm), wrapped with cellophane tape.

7.3.1.3 *Masking Tape*, 13-mm (½-in.) wide.

7.3.1.4 *Plastic Sheet Protector*, clear.

7.3.1.5 *Photographic Reference Standard No. 2*, TNO.<sup>6</sup>

7.3.1.6 *Reflectance Standard*, polished black glass.

7.3.1.7 *Reflectance Standard*, white tile.

NOTE 3—The black reference standard is necessary as the background for this measurement, since the reflectance of black paper is too high. Reflectometers (tristimulus colorimeters), with 0 to 45° geometry, use the Y value.

### 7.3.2 Optional Materials:

7.3.2.1 *China Marker*, black.

<sup>5</sup> Permission to include this test method is provided as a courtesy of NL Chemicals, Wyckoff Mills Rd., Hightstown, NJ 08520.

<sup>6</sup> The TNO Method and photographic reference standard are provided as a courtesy of Verfijnstituut TNO Paint Research Institute TNO, Schoemakerstraat 97, Delft, Nederland. The original source of the photographic reference standard illustrated in Fig. 2 is the Paint Research Institute, TNO. The ASTM numerical rating of chalking shown on the photographic reference standard is opposite to the original TNO scale.

# TAPE CHALK RATING WORKSHEET

## Reflectance Method

Client Name \_\_\_\_\_

Order # \_\_\_\_\_

Radiation to date \_\_\_\_\_

	Measurements	Avg /%	Corrected Value	Rating
Sheet	.	.	.	.
Tape & Sheet	.	.	.	.
1st Set	2nd Set			
1	.	.	.	.
2	.	.	.	.
3	.	.	.	.
4	.	.	.	.
5	.	.	.	.
6	.	.	.	.
7	.	.	.	.
8	.	.	.	.
9	.	.	.	.
10	.	.	.	.
Tape & Sheet	.	.	.	.
Sheet	.	.	.	.

Date \_\_\_\_\_

Inspected by \_\_\_\_\_

NOTE 1—Label sample numbers, apply initial blank tape, and proceed with tape specimens of the samples.

FIG. 3 Example of Worksheet

### 7.3.2.2 Razor Blade.

### 7.3.3 Preparation:

7.3.3.1 Separately mount and apply two 279-mm (11-in.) pieces of masking tape along the right side of the clear plastic sheet cover leaving 32 mm (1¼ in.) of space between the pieces (see Fig. 4).

7.3.3.2 Remove a 50-mm (2-in.) long piece of 13-mm (½-in.) cellulose, pressure-sensitive adhesive tape from the roll; place it across the masking strips, and adhere it to the sheet using a 20-mm (¾-in.) eraser, wrapped with cellophane tape. Label this tape “blank” on the clear plastic sheet cover. A black china marker has been found useful for this purpose.

NOTE 4—The average reflectance measurements of the initial and ending “blank” tapes less the correction value for the clear plastic sheet divided by 100 are used to verify a rating of 10 using Table 1.<sup>7</sup>

### 7.3.4 Procedure:

7.3.4.1 Apply a 50-mm (2-in.) long piece of 13-mm (½-in.) wide tape to the surface being rated. Rub ten times with moderate pressure using the covered eraser, to remove all bubbles and prevent scratching. Remove the tape from the surface and adhere it to the sheet by rubbing with the eraser. Label specimens using a black china marker. Place successive tapes vertically adjacent to previous tapes, separated by 3 mm (⅛ in.). Follow the instructions given in 7.3, and place the final “blank” tape across the masking tape strips and label “blank” on the clear sheet. When completed, use a razor blade to cut along the inside edges of the masking tape, cutting through the adhesive tapes. The removal of the masking tape will leave only the tapes to be measured and evaluated with the sample number of each tape listed on the sheet (see worksheet example in Fig. 4). Before proceeding, check to ensure all sample numbers have been recorded on the sheet.

7.3.4.2 Insert the 13-mm (½ in.) or smaller aperture and calibrate the reflectometer according to the manufacturer’s instructions, setting the reflectometer for zero reflectance using the black reflectance standard and standardizing with the white reflectance standard and record the values. Refer to Test Method E1347 should there be any question on the correct procedure to follow in the calibration of the instrument.

### 7.3.5 Reflectometer Measurements :

7.3.5.1 Leave the transparent tapes mounted on the clear plastic sheet. Remove the black paper that may have been inserted behind the sheet and fold back the unused portion of the sheet. Measure the reflectance of the clear plastic sheet using the black reflectance standard of the instrument (Note 3) as a backing or background and record its value. Move the sheet until the first tape is exposed to the light source with the adhesive side toward the light and the black reflectance standard behind the area being measured, and record the value.

7.3.5.2 Continue this procedure until ten tapes have been measured, then check reflectance values for the white and black standards. If no changes have occurred, proceed with measurements. If values have changed, restandardize and record values before proceeding to the remaining tapes. Following the final tape measurement, record reflectance values of the clear plastic sheet cover, and the white and black reflectance standards.

7.3.5.3 Subtract the mean reflectance value of the sheet from each reading, enter on worksheet form (Fig. 4), or other form used, and determine from Table 1 the chalk rating value of each tape to the nearest 0.5 unit. Record the rating on the worksheet or other form. The worksheet form (Fig. 4), inserted into the plastic sheet protector with a black background gives a clear permanent record of these measurements and evaluations.

7.3.5.4 These tape chalks may also be compared to Photographic Reference Standard No. 2 as an alternative procedure.

<sup>7</sup> Kronos-Titan Table for Chalk Rating from Reflectance Reading using the Transparent Tape Method is provided as a courtesy of Kronos-Titan GmbH, Leverkusen, West Germany. The original source of Table 1 is Kronos-Titan GmbH.

TAPE CHALK RATING WORKSHEET

Reflectance Method

Client Name ABC Company

Order # 164433

Radiation to date 616 MJ/m<sup>2</sup> of uv

**SHEET**  
**BLANK**

	Measurements	Avg/%	Corrected Value	Rating	
Sheet	0.44	0.0045	-	-	
Tape & Sheet	2.38	0.0239	0.0194	10.0	
1st Set	2nd Set				
1	24.20	24.14	0.2380	0.2335	3.0
2	16.46	17.55	0.1664	0.1619	4.5
3	16.91	16.19	0.1618	0.1573	4.5
4	14.65	15.76	0.1484	0.1439	5.0
5	22.14	20.66	0.2103	0.2058	3.5
6	21.33	20.00	0.2046	0.2001	3.5
7	23.11	22.98	0.2268	0.2223	3.0
8	9.54	7.70	0.0862	0.8616	7.5
9	35.79	37.33	0.3619	0.3574	0.5
10	5.86	5.27	0.0520	0.0475	9.0
Tape & Sheet	2.40	0.0239	0.0194	10.0	
Sheet	0.46	0.0045			

**BLANK**  
**SHEET**

Date 6-20-87

Inspected by John R. Doe

NOTE 1—The cutout section is for illustration purposes only. The labeling (china marker) may be removed by rubbing the sheet with a clean tissue or cloth.

FIG. 4 Completed Worksheet

TABLE 1 Chalk Rating from Reflectance Reading<sup>A</sup>

Reflectance Range	Chalk Rating
0–0.038	10
0.0381–0.044	9.5
0.0441–0.054	9.0
0.0541–0.062	8.5
0.0621–0.072	8.0
0.0721–0.082	7.5
0.0821–0.095	7.0
0.0951–0.105	6.5
0.1051–0.120	6.0
0.1201–0.131	5.5
0.1311–0.150	5.0
0.1501–0.165	4.5
0.1651–0.190	4.0
0.1901–0.210	3.5
0.2101–0.235	3.0
0.2351–0.260	2.5
0.2601–0.286	2.0
0.2861–0.310	1.5
0.3101–0.340	1.0
0.3401–0.366	0.5
>0.366	0

<sup>A</sup> This table is based upon a correlation between tape reflectance measurements and visual evaluations of the same tape compared to the photographic standards prepared by Kronos-Titan.<sup>7</sup>

#### 7.4 Test Method D—TNO Type Method:

##### 7.4.1 Materials:

7.4.1.1 *Photographic Reference Standard No. 2* for the determination of chalking, consisting of a photograph of five

strips of tape mounted on a black background, numbered 0, 2, 4, 6, and 8, and varying in this order from black to almost white.

7.4.1.2 *Polyethylene Tape*, transparent, 13 to 25 mm (½ to 1 in.) wide.

7.4.1.3 *Black Velvet*, dull black with a short pile and without a tendency to crush, size approximately 200 by 300 mm (8 by 12 in.), mounted on a flat substrate. Black construction paper may also be used. Place adjacent to the standard for the ratings.

7.4.2 *Procedure*—Apply a piece of tape, 63 to 100 mm (2½ to 4 in.) long to the coating by uniform gentle pressure of the finger, remove the tape, and lay it with the adhesive side on the piece of velvet. Under diffused light compare the tape on the black velvet with Photographic Reference Standard No. 2, and determine which of the five grades most closely matches the whiteness of the adhering pigment. If the degree of chalking is obviously between two adjacent grades, select the intermediate odd number as the chalk rating.

7.4.3 Chalk ratings may also be determined by following the procedures of 7.3.3 and comparing to the values shown in Fig. 1. The use of the worksheet form shown in Fig. 3 and Fig. 4 may be used as a permanent record.

## 8. Report

8.1 A record of the test method used, the rating, panel number, and other pertinent information must be clearly shown on the inspection report for each evaluation.

8.2 The pertinent information should include: date of inspection, date of exposure start, purchase order number of testing organization, duration, remarks about unusual weather, etc., the name of the person making the inspection, and other information agreed upon between the producer and the seller.

## **9. Keywords**

9.1 chalking; evaluation; exterior paint films

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## Standard Test Methods for Measuring Adhesion by Tape Test<sup>1</sup>

This standard is issued under the fixed designation D3359; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—Footnote 5 and 5.2 were corrected editorially in June 2010.

<sup>ε2</sup> NOTE—Footnote 5 and 5.2 were corrected editorially and moved into Note 4 in 5.3 in July 2010.

### 1. Scope\*

1.1 These test methods cover procedures for assessing the adhesion of coating films to metallic substrates by applying and removing pressure-sensitive tape over cuts made in the film.

NOTE 1—This test method has been reported being used to measure adhesion of organic coatings on soft substrates (for example, wood and plastic). Issues with plastic substrates are noted in [Appendix X1](#). A similar test method, ISO 2409, permits tests on soft substrates (for example, wood and plaster). Precision and bias data on the later is lacking. Test Methods D3359 was developed with metal as the substrate and, in the absence of supporting precision and bias data, is so limited.

1.2 Test Method A is primarily intended for use at job sites while Test Method B is more suitable for use in the laboratory. Also, Test Method B is not considered suitable for films thicker than 5 mils (125 $\mu$ m).

NOTE 2—Subject to agreement between the purchaser and the seller, Test Method B can be used for thicker films if wider spaced cuts are employed.

1.3 These test methods are used to establish whether the adhesion of a coating to a substrate is at a generally adequate level. They do not distinguish between higher levels of adhesion for which more sophisticated methods of measurement are required.

NOTE 3—It should be recognized that differences in adherability of the coating surface can affect the results obtained with coatings having the same inherent adhesion.

1.4 This test method is similar in content (but not technically equivalent) to ISO 2409.

1.5 In multicoat systems adhesion failure may occur between coats so that the adhesion of the coating system to the substrate is not determined.

1.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

[D609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products](#)

[D823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels](#)

[D1000 Test Methods for Pressure-Sensitive Adhesive-Coated Tapes Used for Electrical and Electronic Applications](#)

[D1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting](#)

[D2092 Guide for Preparation of Zinc-Coated \(Galvanized\) Steel Surfaces for Painting \(Withdrawn 2008\)<sup>3</sup>](#)

[D2370 Test Method for Tensile Properties of Organic Coatings](#)

[D3330/D3330M Test Method for Peel Adhesion of Pressure-Sensitive Tape](#)

[D3924 Specification for Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials](#)

[D4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser](#)

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).



## 2.2 Other Standard:

**ISO 2409 Paint and Varnishes — Cross-cut test<sup>4</sup>**

## 3. Summary of Test Methods

**3.1 Test Method A**—An X-cut is made through the film to the substrate, pressure-sensitive tape is applied over the cut and then removed, and adhesion is assessed qualitatively on the 0 to 5 scale.

**3.2 Test Method B**—A lattice pattern with either six or eleven cuts in each direction is made in the film to the substrate, pressure-sensitive tape is applied over the lattice and then removed, and adhesion is evaluated by comparison with descriptions and illustrations.

## 4. Significance and Use

**4.1** If a coating is to fulfill its function of protecting or decorating a substrate, it must adhere to it for the expected service life. Because the substrate and its surface preparation (or lack of it) have a drastic effect on the adhesion of coatings, a method to evaluate adhesion of a coating to different substrates or surface treatments, or of different coatings to the same substrate and treatment, is of considerable usefulness in the industry.

**4.2** The limitations of all adhesion methods and the specific limitation of this test method to lower levels of adhesion (see **1.3**) should be recognized before using it. The intra- and inter-laboratory precision of this test method is similar to other widely-accepted tests for coated substrates (for example, Test Method **D2370** and Test Method **D4060**), but this is partly the result of it being insensitive to all but large differences in adhesion. The limited scale of 0 to 5 was selected deliberately to avoid a false impression of being sensitive.

## TEST METHOD A—X-CUT TAPE TEST

## 5. Apparatus and Materials

**5.1 Cutting Tool**—Sharp razor blade, scalpel, knife or other cutting devices. It is of particular importance that the cutting edges be in good condition.

**5.2 Cutting Guide**—Steel or other hard metal straightedge to ensure straight cuts.

**5.3 Tape**—25-mm (1.0-in.) wide semitransparent pressure-sensitive tape with an adhesion strength agreed upon by the supplier and the user is needed. Because of the variability in adhesion strength from batch-to-batch and with time, it is essential that tape from the same batch be used when tests are to be run in different laboratories. If this is not possible the test method should be used only for ranking a series of test coatings.

**NOTE 4**—Permacel P99 tape, previously identified as suitable for this purpose, was withdrawn from manufacture in July 2009. Current supplies of Permacel 99 on the market at this time have a shelf life that runs out in July 2010. Subcommittee D01.23 is assessing alternative tapes and a new interlaboratory study is planned to take place in 2010. Alternative tapes

with specifications similar to that of Permacel P99 tape are available. Users of alternative tapes should check whether the alternative tapes give comparable results to the Permacel P99 tape. If more information is required about the tapes being evaluated in the D01.23 interlaboratory study, please contact the Committee D01 staff manager.

**5.4 Rubber Eraser**, on the end of a pencil.

**5.5 Illumination**—A light source is helpful in determining whether the cuts have been made through the film to the substrate.

## 6. Test Specimens

**6.1** When this test method is used in the field, the specimen is the coated structure or article on which the adhesion is to be evaluated.

**6.2** For laboratory use apply the materials to be tested to panels of the composition and surface conditions on which it is desired to determine the adhesion.

**NOTE 5**—Applicable test panel description and surface preparation methods are given in Practice **D609** and Practices **D1730** and **D2092**.

**NOTE 6**—Coatings should be applied in accordance with Practice **D823**, or as agreed upon between the purchaser and the seller.

**NOTE 7**—If desired or specified, the coated test panels may be subjected to a preliminary exposure such as water immersion, salt spray, or high humidity before conducting the tape test. The conditions and time of exposure will be governed by ultimate coating use or shall be agreed upon between the purchaser and seller.

## 7. Procedure

**7.1** Select an area free of blemishes and minor surface imperfections. For tests in the field, ensure that the surface is clean and dry. Extremes in temperature or relative humidity may affect the adhesion of the tape or the coating.

**7.1.1** For specimens which have been immersed: After immersion, clean and wipe the surface with an appropriate solvent which will not harm the integrity of the coating. Then dry or prepare the surface, or both, as agreed upon between the purchaser and the seller.

**7.2** Make two cuts in the film each about 40 mm (1.5 in.) long that intersect near their middle with a smaller angle of between 30 and 45°. When making the incisions, use the straightedge and cut through the coating to the substrate in one steady motion.

**7.3** Inspect the incisions for reflection of light from the metal substrate to establish that the coating film has been penetrated. If the substrate has not been reached make another X in a different location. Do not attempt to deepen a previous cut as this may affect adhesion along the incision.

**7.4** At each day of testing, before initiation of testing, remove two complete laps of the pressure-sensitive tape from the roll and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 75 mm (3 in.) long.

**7.5** Place the center of the tape at the intersection of the cuts with the tape running in the same direction as the smaller angles. Smooth the tape into place by finger in the area of the incisions and then rub firmly with the eraser on the end of a pencil. The color under the transparent tape is a useful indication of when good contact has been made.

<sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

7.6 Within  $90 \pm 30$  s of application, remove the tape by seizing the free end and pulling it off rapidly (not jerked) back upon itself at as close to an angle of  $180^\circ$  as possible.

7.7 Inspect the X-cut area for removal of coating from the substrate or previous coating and rate the adhesion in accordance with the following scale:

- 5A No peeling or removal,
- 4A Trace peeling or removal along incisions or at their intersection,
- 3A Jagged removal along incisions up to 1.6 mm ( $\frac{1}{16}$  in.) on either side,
- 2A Jagged removal along most of incisions up to 3.2 mm ( $\frac{1}{8}$  in.) on either side,
- 1A Removal from most of the area of the X under the tape, and
- 0A Removal beyond the area of the X.

7.8 Repeat the test in two other locations on each test panel. For large structures make sufficient tests to ensure that the adhesion evaluation is representative of the whole surface.

7.9 After making several cuts examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone before using again. Discard cutting tools that develop nicks or other defects that tear the film.

## 8. Report

8.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred that is, between first coat and substrate, between first and second coat, etc.

8.2 For field tests report the structure or article tested, the location and the environmental conditions at the time of testing.

8.3 For test panels report the substrate employed, the type of coating, the method of cure, and the environmental conditions at the time of testing.

8.4 If the adhesion strength of the tape has been determined in accordance with Test Methods **D1000** or **D3330/D3330M**, report the results with the adhesion rating(s). If the adhesion strength of the tape has not been determined, report the specific tape used and its manufacturer.

8.5 If the test is performed after immersion, report immersion conditions and method of sample preparation.

## 9. Precision and Bias<sup>5</sup>

9.1 In an interlaboratory study of this test method in which operators in six laboratories made one adhesion measurement on three panels each of three coatings covering a wide range of adhesion, the within-laboratories standard deviation was found to be 0.33 and the between-laboratories 0.44. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Provided adhesion is uniform over a large surface, results obtained by the same operator should be considered suspect if they differ by more than 1 rating unit for two measurements.

9.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by different operators should be considered suspect if they differ by more than 1.5 rating units.

9.2 Bias cannot be established for these test methods.

## TEST METHOD B—CROSS-CUT TAPE TEST

### 10. Apparatus and Materials

10.1 *Cutting Tool*<sup>6</sup>—Sharp razor blade, scalpel, knife or other cutting device having a cutting edge angle between  $15^\circ$  and  $30^\circ$  that will make either a single cut or several cuts at once. It is of particular importance that the cutting edge or edges be in good condition.

10.2 *Cutting Guide*—If cuts are made manually (as opposed to a mechanical apparatus) a steel or other hard metal straight-edge or template to ensure straight cuts.

10.3 *Rule*—Tempered steel rule graduated in 0.5 mm for measuring individual cuts.

10.4 *Tape*, as described in **5.3**.

10.5 *Rubber Eraser*, on the end of a pencil.

10.6 *Illumination*, as described in **5.5**.

10.7 *Magnifying Glass*—An illuminated magnifier to be used while making individual cuts and examining the test area.

### 11. Test Specimens

11.1 Test specimens shall be as described in Section **6**. It should be noted, however, that multitip cutters<sup>7</sup> provide good results only on test areas sufficiently plane that all cutting edges contact the substrate to the same degree. Check for flatness with a straight edge such as that of the tempered steel rule (**10.3**).

### 12. Procedure

12.1 Where required or when agreed upon, subject the specimens to a preliminary test before conducting the tape test (see **Note 5**). After drying or testing the coating, conduct the tape test at room temperature as defined in Specification **D3924**, unless **D3924** standard temperature is required or agreed.

12.1.1 For specimens which have been immersed: After immersion, clean and wipe the surface with an appropriate solvent which will not harm the integrity of the coating. Then dry or prepare the surface, or both, as agreed upon between the purchaser and the seller.

12.2 Select an area free of blemishes and minor surface imperfections, place on a firm base, and under the illuminated magnifier, make parallel cuts as follows:

<sup>6</sup> Multiblade cutters are available from a few sources that specialize in testing equipment for the paint industry.

<sup>7</sup> The sole source of supply of the multitip cutter for coated pipe surfaces known to the committee at this time is Paul N. Gardner Co., 316 NE First St., Pompano Beach, FL 33060. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1008. Contact ASTM Customer Service at service@astm.org.

12.2.1 For coatings having a dry film thickness up to and including 2.0 mils (50 μm) space the cuts 1 mm apart and make eleven cuts unless otherwise agreed upon.

12.2.2 For coatings having a dry film thickness between 2.0 mils (50 μm) and 5 mils (125 μm), space the cuts 2 mm apart and make six cuts. For films thicker than 5 mils (125 μm), use Test Method A.<sup>8</sup>

12.2.3 Make all cuts about 20 mm (¾ in.) long. Cut through the film to the substrate in one steady motion using just sufficient pressure on the cutting tool to have the cutting edge reach the substrate. When making successive single cuts with the aid of a guide, place the guide on the uncut area.

12.3 After making the required cuts brush the film lightly with a soft brush or tissue to remove any detached flakes or ribbons of coatings.

12.4 Examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone. Make the additional number of cuts at 90° to and centered on the original cuts.

12.5 Brush the area as before and inspect the incisions for reflection of light from the substrate. If the metal has not been reached make another grid in a different location.

12.6 At each day of testing, before initiation of testing, remove two complete laps of tape and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 75 mm (3 in.) long.

12.7 Place the center of the tape over the grid and in the area of the grid smooth into place by a finger. To ensure good contact with the film rub the tape firmly with the eraser on the end of a pencil. The color under the tape is a useful indication of when good contact has been made.

12.8 Within 90 ± 30 s of application, remove the tape by seizing the free end and rapidly (not jerked) back upon itself at as close to an angle of 180° as possible.

12.9 Inspect the grid area for removal of coating from the substrate or from a previous coating using the illuminated magnifier. Rate the adhesion in accordance with the following scale illustrated in Fig. 1:

- 5B The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
- 4B Small flakes of the coating are detached at intersections; less than 5 % of the area is affected.
- 3B Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
- 2B The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
- 1B The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.
- 0B Flaking and detachment worse than Grade 1.

12.10 Repeat the test in two other locations on each test panel.

CLASSIFICATION OF ADHESION TEST RESULTS		
CLASSIFICATION	PERCENT AREA REMOVED	SURFACE OF CROSS-CUT AREA FROM WHICH FLAKING HAS OCCURRED FOR SIX PARALLEL CUTS AND ADHESION RANGE BY PERCENT
5B	0% None	
4B	Less than 5%	
3B	5 – 15%	
2B	15 – 35%	
1B	35 – 65%	
0B	Greater than 65%	

FIG. 1 Classification of Adhesion Test Results

### 13. Report

13.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred, that is, between first coat and substrate, between first and second coat, etc.

13.2 Report the substrate employed, the type of coating and the method of cure.

13.3 If the adhesion strength has been determined in accordance with Test Methods D1000 or D3330/D3330M, report the results with the adhesion rating(s). If the adhesion strength of the tape has not been determined, report the specific tape used and its manufacturer.

13.4 If the test is performed after immersion, report immersion conditions and method of sample preparation.

### 14. Precision and Bias<sup>5</sup>

14.1 On the basis of two interlaboratory tests of this test method in one of which operators in six laboratories made one adhesion measurement on three panels each of three coatings covering a wide range of adhesion and in the other operators in six laboratories made three measurements on two panels each of four different coatings applied over two other coatings, the

<sup>8</sup> Test Method B has been used successfully by some people on coatings greater than 5 mils (0.13 mm) by spacing the cuts 5 mm apart. However, the precision values given in 14.1 do not apply as they are based on coatings less than 5 mils (0.13 mm) in thickness.



pooled standard deviations for within- and between-laboratories were found to be 0.37 and 0.7. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

14.1.1 *Repeatability*—Provided adhesion is uniform over a large surface, results obtained by the same operator should be considered suspect if they differ by more than one rating unit for two measurements.

14.1.2 *Reproducibility*—Two results, each the mean of duplicates or triplicates, obtained by different operators should be considered suspect if they differ by more than two rating units.

14.2 Bias cannot be established for these test methods.

## 15. Keywords

15.1 adhesion; crosscut adhesion test method; tape; tape adhesion test method; X-cut adhesion test method

## APPENDIX

### (Nonmandatory Information)

#### X1. COMMENTARY

##### X1.1 Introduction

X1.1.1 Given the complexities of the adhesion process, can adhesion be measured? As Mittal (1)<sup>9</sup> has pointed out, the answer is both yes and no. It is reasonable to state that at the present time no test exists that can precisely assess the actual physical strength of an adhesive bond. But it can also be said that it is possible to obtain an indication of relative adhesion performance.

X1.1.2 Practical adhesion test methods are generally of two types: “*implied*” and “*direct*.” “*Implied*” tests include indentation or scribe techniques, rub testing, and wear testing. Criticism of these tests arises when they are used to quantify the strength of adhesive bonding. But this, in fact, is not their purpose. An “*implied*” test should be used to assess coating performance under actual service conditions. “*Direct*” measurements, on the other hand, are intended expressly to measure adhesion. Meaningful tests of this type are highly sought after, primarily because the results are expressed by a single discrete quantity, the force required to rupture the coating/substrate bond under prescribed conditions. Direct tests include the Hesiometer and the Adherometer (2). Common methods which approach the direct tests are peel, lap-shear, and tensile tests.

##### X1.2 Test Methods

X1.2.1 In practice, numerous types of tests have been used to attempt to evaluate adhesion by inducing bond rupture by different modes. Criteria deemed essential for a test to warrant large-scale acceptance are: use of a straightforward and unambiguous procedure; relevance to its intended application; repeatability and reproducibility; and quantifiability, including a meaningful rating scale for assessing performance.

X1.2.2 Test methods used for coatings on metals are: peel adhesion or “tape testing;” Gardner impact flexibility testing; and adhesive joint testing including shear (lap joint) and direct tensile (butt joint) testing. These tests do not strictly meet all the criteria listed, but an appealing aspect of these tests is that

in most cases the equipment/instrumentation is readily available or can be obtained at reasonable cost.

X1.2.3 A wide diversity of tests methods have been developed over the years that measure aspects of adhesion (1-5). There generally is difficulty, however, in relating these tests to basic adhesion phenomena.

##### X1.3 The Tape Test

X1.3.1 By far the most prevalent test for evaluating coating “adhesion” is the tape-and-peel test, which has been used since the 1930’s. In its simplest version a piece of adhesive tape is pressed against the paint film and the resistance to and degree of film removal observed when the tape is pulled off. Since an intact film with appreciable adhesion is frequently not removed at all, the severity of the test is usually enhanced by cutting into the film a figure X or a cross hatched pattern, before applying and removing the tape. Adhesion is then rated by comparing film removed against an established rating scale. If an intact film is peeled cleanly by the tape, or if it debonds just by cutting into it without applying tape, then the adhesion is rated simply as poor or very poor, a more precise evaluation of such films not being within the capability of this test.

X1.3.2 The current widely-used version was first published in 1974; two test methods are covered in this standard. Both test methods are used to establish whether the adhesion of a coating to a substrate is at an adequate level; however they do not distinguish between higher levels of adhesion for which more sophisticated methods of measurement are required. Major limitations of the tape test are its low sensitivity, applicability only to coatings of relatively low bond strengths, and non-determination of adhesion to the substrate where failure occurs within a single coat, as when testing primers alone, or within or between coats in multicoat systems. For multicoat systems where adhesion failure may occur between or within coats, the adhesion of the coating system to the substrate is not determined.

X1.3.3 Repeatability within one rating unit is generally observed for coatings on metals for both methods, with reproducibility of one to two units. The tape test enjoys widespread popularity and is viewed as “simple” as well as low

<sup>9</sup> The boldface numbers in parentheses refer to the list of references at the end of this test method.

in cost. Applied to metals, it is economical to perform, lends itself to job site application, and most importantly, after decades of use, people feel comfortable with it.

X1.3.4 When a flexible adhesive tape is applied to a coated rigid substrate surface and then removed, the removal process has been described in terms of the “peel phenomenon,” as illustrated in Fig. X1.1.

X1.3.5 Peeling begins at the “toothed” leading edge (at the right) and proceeds along the coating adhesive/interface or the coating/substrate interface, depending on the relative bond strengths. It is assumed that coating removal occurs when the tensile force generated along the latter interface, which is a function of the rheological properties of the backing and adhesive layer materials, is greater than the bond strength at the coating-substrate interface (or cohesive strength of the coating). In actuality, however, this force is distributed over a discrete distance (O-A) in Fig. X1.1, which relates directly to the properties described, not concentrated at a point (O) in Fig. X1.1 as in the theoretical case—though the tensile force is greatest at the origin for both. A significant compressive force arises from the response of the tape backing material to being stretched. Thus both tensile and compressive forces are involved in adhesion tape testing.

X1.3.6 Close scrutiny of the tape test with respect to the nature of the tape employed and certain aspects of the procedure itself reveal several factors, each or any combination of which can dramatically affect the results of the test as discussed (6).

#### X1.4 Peel Adhesion Testing on Plastic Substrates

X1.4.1 Tape tests have been criticized when used for substrates other than metal, such as plastics. The central issues are that the test on plastics lacks reproducibility and does not relate to the intended application. Both concerns are well founded: poor precision is a direct result of several factors intrinsic to the materials employed and the procedure itself. More importantly, in this instance the test is being applied beyond its intended scope. These test methods were designed

for relatively ductile coatings applied to metal substrates, not for coatings (often brittle) applied to plastic parts (7). The unique functional requirements of coatings on plastic substrates cause the usual tape tests to be unsatisfactory for measuring adhesion performance in practice.

#### X1.5 The Tape Controversy

X1.5.1 With the withdrawal from commerce of the tape specified originally, 3M No. 710, current test methods no longer identify a specific tape. Differences in tapes used can lead to different results as small changes in backing stiffness and adhesive rheology cause large changes in the tension area. Some commercial tapes are manufactured to meet minimum standards. A given lot may surpass these standards and thus be suitable for general market distribution; however, such a lot may be a source of serious and unexpected error in assessing adhesion. One commercially available tape test kit had included a tape with adhesion strength variations of up to 50 % claimed by the manufacturer. Also, because tapes change on storage, bond strengths of the tape may change over time (7, 8).

X1.5.2 While there are tapes available that appear to deliver consistent performance, a given tape does not adhere equally well to all coatings. For example, when the peel removal force of the tape (from the coating) used earlier by Task Group D01.23.10 to establish precision of the method, by 3M No. 710 was examined with seven different electromagnetic interference/radio frequency interference (EMI/RFI) coatings, it was found that, while peel was indeed consistent for a given coating, the value varied by 25 % between the highest and lowest ratings among coatings. Several factors that contribute to these differences include coating composition and topology: as a result, no single tape is likely to be suitable for testing all coatings. Further, the tape test does not give an absolute value for the force required for bond rupture, but serves only as an indicator that some minimum value for bond strength was met or exceeded (7, 8).

#### X1.6 Procedural Problems

X1.6.1 The tape test is operator intensive. By design it was made as simple as possible to perform, and requires a minimum of specialized equipment and materials that must meet certain specifications. The accuracy and precision depend largely upon the skill of the operator and the operator’s ability to perform the test in a consistent manner. Key steps that directly reflect the importance of operator skill include the angle and rate of tape removal and the visual assessment of the tested sample. It is not unexpected that different operators might obtain different results (7, 8).

X1.6.2 *Peel Angle and Rate:* The standard requires that the free end of the tape be removed rapidly at as close to a 180° angle as possible. If the peel angle and rate vary, the force required to remove the tape can change dramatically. Nearly linear increases were observed in peel force approaching 100 % as peel angle was changed from 135 to 180, and similar large differences can be expected in peel force as peel rate

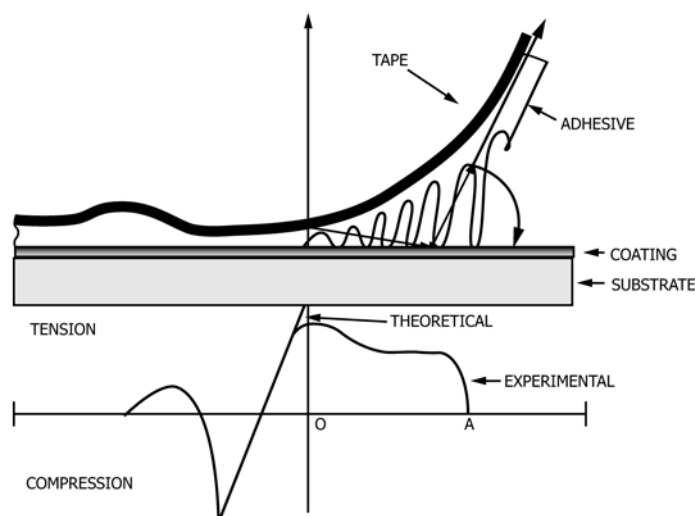


FIG. X1.1 Peel Profile (6)

varies. These effects are related as they reflect certain rheological properties of the backing and adhesive that are molecular in origin. Variation in pull rate and peel angle can effect large differences in test values and must be minimized to assure reproducibility (9).

**X1.6.3 Visual Assessment:** The final step in the test is visual assessment of the coating removed from the specimen, which is subjective in nature, so that the coatings can vary among individuals evaluating the same specimen (9).

**X1.6.3.1 Performance** in the tape test is based on the amount of coating removed compared to a descriptive scale. The exposure of the substrate can be due to factors other than coating adhesion, including that arising from the requirement that the coating be cut (hence the synonym “cross-hatch adhesion test”). Justification for the cutting step is reasonable as cutting provides a free edge from which peeling can begin without having to overcome the cohesive strength of the coating layer.

**X1.6.3.2** Cutting might be suitable for coatings applied to metal substrates, but for coatings applied to plastics or wood, the process can lead to a misleading indication of poor adhesion due to the unique interfacial zone. For coatings on soft substrates, issues include how deep should this cut penetrate, and is it possible to cut only to the interface?

**X1.6.3.3** In general, if adhesion test panels are examined microscopically, it is often clearly evident that the coating removal results from substrate failure at or below the interface, and not from the adhesive failure between the coating and the substrate. Cohesive failure within the coating film is also frequently observed. However, with the tape test, failures within the substrate or coating layers are rare because the tape adhesive is not usually strong enough to exceed the cohesive

strengths of normal substrates and organic coatings. Although some rather brittle coatings may exhibit cohesive failure, the tape test adhesion method does not make provision for giving failure locality (7, 8).

**X1.6.4** Use of the test method in the field can lead to variation in test results due to temperature and humidity changes and their effect upon tape, coating and substrate.

**X1.6.5** Test Method B has been used successfully, without affecting adhesion test results, by some coil coating users on coatings up to and including 2.0 mils (50 μm) by spacing the cuts 2 mm apart. While this may be an agreement between purchaser and seller, the precision values given in 14.1 do not apply, as they are based on cuts 1 mm apart.

**X1.6.6** Some have found that the use of a suitable mechanical device is helpful in minimizing some of the variables in placing the tape onto the coatings (see 7.5 and 12.7) and removing the tape from the coatings (see 7.6 and 12.8) which maintains consistent pressure on the tape during application and ensures a 180° pull off.<sup>10</sup>

## X1.7 Conclusion

**X1.7.1** All the issues aside, if these test methods are used within the Scope Section and are performed carefully, some insight into the approximate, relative level of adhesion can be gained.

<sup>10</sup> The sole source of supply of a suitable mechanical device for laying down and removal of tape known to the committee at this time is ReliaPull, a registered trademark of Random Logic LLC, manufactured by Random Logic LLC, Cincinnati, OH 45245. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

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## SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D3359 - 08) that may impact the use of this standard. (Approved June 1, 2009.)

(1) X1.6.6 was added to Appendix X1.

(2) Footnote 10 was added to identify a source of equipment.

Committee D01 has identified the location of selected changes to this standard since the last issue (D3359 - 07) that may impact the use of this standard. (Approved February 1, 2008.)

(1) X1.6.5 was added to Appendix X1.

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# Standard Test Methods for Water Vapor Transmission of Materials<sup>1</sup>

This standard is issued under the fixed designation E96/E96M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

## 1. Scope

1.1 These test methods cover the determination of water vapor transmission (WVT) of materials through which the passage of water vapor may be of importance, such as paper, plastic films, other sheet materials, fiberboards, gypsum and plaster products, wood products, and plastics. The test methods are limited to specimens not over 1¼ in. [32 mm] in thickness except as provided in Section 9. Two basic methods, the Desiccant Method and the Water Method, 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. Agreement should not be expected between results obtained by different methods. The method should be selected that more nearly approaches the conditions of use.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard. However, derived results can be converted from one system to the other using appropriate conversion factors (see Table 1).

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

**C168 Terminology Relating to Thermal Insulation**

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee C16 on Thermal Insulation and are the direct responsibility of Subcommittee C16.33 on Insulation Finishes and Moisture.

Current edition approved Oct. 15, 2014. Published January 2015. Originally approved in 1953. Last previous edition approved in 2013 as E96–13. DOI: 10.1520/E0096\_E0096M-14.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

**E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods**

**D449 Specification for Asphalt Used in Dampproofing and Waterproofing**

**D2301 Specification for Vinyl Chloride Plastic Pressure-Sensitive Electrical Insulating Tape**

**E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method**

## 3. Terminology

3.1 Definitions of terms used in this standard will be found in Terminology C168, from which the following is quoted:

*“water vapor permeability—*the time rate of water vapor transmission through unit area of flat material of unit thickness induced by unit vapor pressure difference between two specific surfaces, under specified temperature and humidity conditions.

*Discussion—*Permeability is a property of a material, but the permeability of a body that performs like a material may be used. Permeability is the arithmetic product of permeance and thickness.

*water vapor permeance—*the time rate of water vapor transmission through unit area of flat material or construction induced by unit vapor pressure difference between two specific surfaces, under specified temperature and humidity conditions.

*Discussion—*Permeance is a performance evaluation and not a property of a material.

3.2 *water vapor transmission rate—*the steady water vapor flow in unit time through unit area of a body, normal to specific parallel surfaces, under specific conditions of temperature and humidity at each surface.”

## 4. Summary of Test Methods

4.1 In the Desiccant Method the test specimen is sealed to the open mouth of a test dish containing a desiccant, and the assembly placed in a controlled atmosphere. Periodic weighings determine the rate of water vapor movement through the specimen into the desiccant.



**TABLE 1 Metric Units and Conversion Factors<sup>A,B</sup>**

Multiply	by	To Obtain (for the same test condition)
<i>WVT</i>		
g/h·m <sup>2</sup>	1.43	grains/h·ft <sup>2</sup>
grains/h·ft <sup>2</sup>	0.697	g/h·m <sup>2</sup>
<i>Permeance</i>		
g/Pa·s·m <sup>2</sup>	1.75 × 10 <sup>7</sup>	1 Perm (inch-pound)
1 Perm (inch-pound)	5.72 × 10 <sup>-8</sup>	g/Pa·s·m <sup>2</sup>
<i>Permeability</i>		
g/Pa·s·m	6.88 × 10 <sup>8</sup>	1 Perm inch
1 Perm inch	1.45 × 10 <sup>-9</sup>	g/Pa·s·m

<sup>A</sup> These units are used in the construction trade. Other units may be used in other standards.

<sup>B</sup> All conversions of mm Hg to Pa are made at a temperature of 0°C.

4.2 In the Water Method, the dish contains distilled water, and the weighings determine the rate of vapor movement through the specimen from the water to the controlled atmosphere. The vapor pressure difference is nominally the same in both methods except in the variation, with extremes of humidity on opposite sides.

## 5. Significance and Use

5.1 The purpose of these tests is to obtain, by means of simple apparatus, reliable values of water vapor transfer through permeable and semipermeable materials, expressed in suitable units. These values are for use in design, manufacture, and marketing. A permeance value obtained under one set of test conditions may not indicate the value under a different set of conditions. For this reason, the test conditions should be selected that most closely approach the conditions of use. While any set of conditions may be used and those conditions reported, standard conditions that have been useful are shown in [Appendix X1](#).

## 6. Apparatus

6.1 *Test Dish*—The test dish shall be of any noncorroding material, impermeable to water or water vapor. It may be of any shape. Light weight is desirable. A large, shallow dish is preferred, but its size and weight are limited when an analytical balance is chosen to detect small weight changes. The mouth of the dish shall be as large as practical and at least 4.65 in.<sup>2</sup> [3000 mm<sup>2</sup>]. The desiccant or water area shall be not less than the mouth area except if a grid is used, as provided in [12.1](#), its effective area shall not exceed 10 % of the mouth area. An external flange or ledge around the mouth, to which the specimen may be attached, is useful when shrinking or warping occurs. When the specimen area is larger than the mouth area, this overlay upon the ledge is a source of error, particularly for thick specimens. This overlay material should be masked as described in [10.1](#) so that the mouth area defines the test area. The overlay material results in a positive error, indicating excessive water vapor transmission. The magnitude of the error is a complex function of the thickness, ledge width, mouth area, and possibly the permeability. This error is discussed by Joy and Wilson ([1](#))<sup>3</sup> (see [13.4.3](#)). This type of error should be

limited to about 10 to 12 %. For a thick specimen the ledge should not exceed ¾ in. [19 mm] for a 10-in. [254-mm] or larger mouth (square or circular) or ⅛ in. [3 mm] for a 5-in. [127-mm] mouth (square or circular). For a 3-in. [76-mm] mouth (square or circular) the ledge should not exceed 0.11 in. [2.8 mm] wide. An allowable ledge may be interpolated for intermediate sizes or calculated according to Joy and Wilson. ([1](#)) A rim around the ledge ([Fig. X2.1](#)) may be useful. If a rim is provided, it shall be not more than ¼ in. [6 mm] higher than the specimen as attached. Different depths may be used for the Desiccant Method and Water Method, but a ¾-in. [19-mm] depth (below the mouth) is satisfactory for either method.

6.2 *Test Chamber*—The room or cabinet where the assembled test dishes are to be placed shall have a controlled temperature (see [Note 1](#)) and relative humidity. Some standard test conditions that have been useful are given in [Appendix X1](#). The temperature chosen shall be determined according to the desired application of the material to be tested (see [Appendix X1](#)). The relative humidity shall be maintained at 50 ± 2 %, except where extremes of humidities are desired, when the conditions shall be 100 ± 1.8°F [38 ± 1°C] and 90 ± 2 % relative humidity. Both temperature and relative humidity shall be measured frequently<sup>4</sup> or preferably recorded continuously. Air shall be continuously circulated throughout the chamber, with a velocity sufficient to maintain uniform conditions at all test locations. The air velocity over the specimen shall be between 0.066 and 1 ft/s [0.02 and 0.3 m·s<sup>-1</sup>]. Suitable racks shall be provided on which to place the test dishes within the test chamber.

**NOTE 1**—Simple temperature control by heating alone is usually made possible at 90°F [32°C]. However, it is very desirable to enter the controlled space, and a comfortable temperature is more satisfactory for that arrangement. Temperatures of 73.4°F [23°C] and 80°F [26.7°C] are in use and are satisfactory for this purpose. With cyclic control, the average test temperature may be obtained from a sensitive thermometer in a mass of dry sand. The temperature of the chamber walls facing a specimen over water should not be cooler than the water to avoid condensation on the test specimen.

6.3 *Balance and Weights*—The balance shall be sensitive to a change smaller than 1 % of the weight change during the period when a steady state is considered to exist. The weights used shall be accurate to 1 % of the weight change during the steady-state period ([Note 2](#)). A light wire sling may be substituted for the usual pan to accommodate a larger and heavier load.

6.4 *Thickness-Measuring Gage*—The nominal thickness of the specimen shall be determined using a thickness-measuring gage with an accuracy of ±1 % of the reading or 0.0001 in. [0.0025 mm], whichever is greater.

**NOTE 2**—For example: 1-perm [57 ng·Pa<sup>-1</sup>·s<sup>-1</sup>·m<sup>-2</sup>] specimen 10 in. [254 mm] square at 80°F [26.7°C] passes 8.6 grains or 0.56 g/day. In 18 days of steady state, the transfer is 10 g. For this usage, the balance must have a sensitivity of 1 % of 10 g or 0.1 g and the weights must be accurate to 0.1 g. If, however, the balance has a sensitivity of 0.2 g or the weights are no better than 0.2 g, the requirements of this paragraph can be met by

<sup>3</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

<sup>4</sup> The minimum acceptable is to perform this measurement each time the sample is weighed.

continuing the steady state for 36 days. An analytical balance that is much more sensitive will permit more rapid results on specimens below 1 perm [ $57 \text{ ng} \cdot \text{Pa}^{-1} \cdot \text{s}^{-1} \cdot \text{m}^{-2}$ ] when the assembled dish is not excessively heavy.

## 7. Materials

### 7.1 Desiccant and Water:

7.1.1 For the Desiccant Method, anhydrous calcium chloride in the form of small lumps that will pass a No. 8 [2.36-mm] sieve, and free of fines that will pass a No. 30 [600- $\mu\text{m}$ ] sieve, shall be used (Note 3). It shall be dried at 400°F [200°C] before use.

NOTE 3—If  $\text{CaCl}_2$  will react chemically on the specimen, an adsorbing desiccant such as silica gel, activated at 400°F [200°C], may be used; but the moisture gain by this desiccant during the test must be limited to 4 %.

7.1.2 For the Water Method, distilled water shall be used in the test dish.

7.2 Sealant—The sealant used for attaching the specimen to the dish, in order to be suitable for this purpose, must be highly resistant to the passage of water vapor (and water). It must not lose weight to, or gain weight from, the atmosphere in an amount, over the required period of time, that would affect the test result by more than 2 %. It must not affect the vapor pressure in a water-filled dish. Molten asphalt or wax is required for permeance tests below 4 perms [ $230 \text{ ng} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ ]. Sealing methods are discussed in Appendix X2.

## 8. Sampling

8.1 The material shall be sampled in accordance with standard methods of sampling applicable to the material under test. The sample shall be of uniform thickness. If the material is of nonsymmetrical construction, the two faces shall be designated by distinguishing marks (for example, on a one-side-coated sample, “I” for the coated side and “II” for the uncoated side).

## 9. Test Specimens

9.1 Test specimens shall be representative of the material tested. When a product is designed for use in only one position, three specimens shall be tested by the same method with the vapor flow in the designated direction. When the sides of a product are indistinguishable, three specimens shall be tested by the same method. When the sides of a product are different and either side may face the vapor source, four specimens shall be tested by the same method, two being tested with the vapor flow in each direction and so reported.

9.2 A slab, produced and used as a laminate (such as a foamed plastic with natural “skins”) may be tested in the thickness of use. Alternatively, it may be sliced into two or more sheets, each being separately tested and so reported as provided in 9.4, provided also, that the “overlay upon the cup ledge” (6.1) of any laminate shall not exceed  $\frac{1}{8}$  in. [3 mm].

9.3 When the material as used has a pitted or textured surface, the tested thickness shall be that of use. When it is homogeneous, however, a thinner slice of the slab may be tested as provided in 9.4.

9.4 In either case (9.2 or 9.3), the tested overall thickness, if less than that of use, shall be at least five times the sum of the

maximum pit depths in both its faces, and its tested permeance shall be not greater than 5 perms [ $\approx 300 \text{ ng} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ ].

9.5 For homogeneous (not laminated) materials with thickness greater than  $\frac{1}{2}$  in., the overall nominal thickness of each specimen shall be measured with an accuracy of  $\pm 1$  % of the reading at the center of each quadrant and the results averaged.

9.6 When testing any material with a permeance less than 0.05 perms [ $3 \text{ ng} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ ] or when testing a low permeance material that may be expected to lose or gain weight throughout the test (because of evaporation or oxidation), it is strongly recommended that an additional specimen, or “dummy,” be tested exactly like the others, except that no desiccant or water is put in the dish. Failure to use this dummy specimen to establish modified dish weights may significantly increase the time required to complete the test. Because time to reach equilibrium of water permeance increases as the square of thickness, thick, particularly hygroscopic, materials may take as long as 60 days to reach equilibrium conditions.

## 10. Attachment of Specimen to Test Dish

10.1 Attach the specimen to the dish by sealing (and clamping if desired) in such a manner that the dish mouth defines the area of the specimen exposed to the vapor pressure in the dish. If necessary, mask the specimen top surface, exposed to conditioned air so that its exposure duplicates the mouth shape and size and is directly above it. A template is recommended for locating the mask. Thoroughly seal the edges of the specimen to prevent the passage of vapor into, or out of, or around the specimen edges or any portion thereof. The same assurance must apply to any part of the specimen faces outside their defined areas. Suggested methods of attachment are described in Appendix X2.

NOTE 4—In order to minimize the risk of condensation on the interior surface of the sample when it is placed in the chamber, the temperature of the water prior to preparation of the test specimen should be within  $\pm 2^\circ\text{F}$  [ $\pm 1^\circ\text{C}$ ] of the test condition.

## 11. Procedure for Desiccant Method

11.1 Fill the test dish with desiccant within  $\frac{1}{4}$  in. [6 mm] of the specimen. Leave enough space so that shaking of the dish, which must be done at each weighing, will mix the desiccant.

11.2 Attach the specimen to the dish (see 10.1) and place it in the controlled chamber, specimen up, weighing it at once. (This weight may be helpful to an understanding of the initial moisture in the specimen.)

11.3 Weigh the dish assembly periodically, often enough to provide eight or ten data points during the test. A data point is the weight at a particular time. The time that the weight is made should be recorded to a precision of approximately 1 % of the time span between successive weighing. Thus, if weighings are made every hour, record the time to the nearest 30 s; if recordings are made every day, a time to the nearest 15 min would be allowed. At first the weight may change rapidly; later a steady state will be reached where the rate of change is substantially constant. Weighings should be accomplished without removal of the test dishes from the controlled atmosphere, but if removal is prescribed necessary, the time the

specimens are kept at different conditions, temperature or relative humidity, or both, should be kept to a minimum. When results of water vapor transmission are expected to be less than 0.05 perm [3 ng·m<sup>-2</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>], a dummy specimen is strongly recommended. Such a dummy specimen should be attached to an empty cup in the normal manner. The environmental effects of temperature variation and buoyancy variability due to barometric pressure fluctuation can be arithmetically tared out of the weighing values. This precaution permits earlier and more reliable achievement of equilibrium conditions. Analyze the results as prescribed in 13.1.

11.4 Terminate the test or change the desiccant before the water added to the desiccant exceeds 10 % of its starting weight. This limit cannot be exactly determined and judgement is required. The desiccant gain may be more or less than the dish weight-gain when the moisture content of the specimen has changed.

NOTE 5—The WVT of some materials (especially wood) may depend on the ambient relative humidity immediately before the test. An apparent hysteresis results in higher WVT if the prior relative humidity was above the test condition and vice versa. It is therefore recommended that specimens of wood and paper products be conditioned to constant weight in a 50 % relative humidity atmosphere before they are tested. Some specimens may be advantageously preconditioned to minimize the moisture that the specimen will give up to the desiccant. This applies when the specimen is likely to have high moisture content or when it is coated on the top (vapor source) side.

## 12. Procedure for Water Method

12.1 Fill the test dish with distilled water to a level  $\frac{3}{4} \pm \frac{1}{4}$  in. [19 ± 6 mm] from the specimen. The air space thus allowed has a small vapor resistance, but it is necessary in order to reduce the risk of water touching the specimen when the dish is handled. Such contact invalidates a test on some materials such as paper, wood, or other hygroscopic materials. The water depth shall be not less than  $\frac{1}{8}$  in. [3 mm] to ensure coverage of the dish bottom throughout the test. However, if the dish is of glass, its bottom must be visibly covered at all times but no specific depth is required. Water surges may be reduced by placing a grid of light noncorroding material in the dish to break the water surface. This grid shall be at least  $\frac{1}{4}$  in. [6 mm] below the specimen, and it shall not reduce the water surface by more than 10 %.

NOTE 6—For the Water Method, baking the empty dish and promptly coating its mouth with sealant before assembly is recommended. The water may be added most conveniently after the specimen is attached, through a small sealable hole in the dish above the water line.

12.2 Attach the specimen to the dish (see 10.1). Some specimens are likely to warp and break the seal during the test. The risk is reduced by preconditioning the specimen, and by clamping it to the dish ledge (if one is provided).

12.3 Weigh the dish assembly and place it in the controlled chamber on a true horizontal surface. Follow the procedure given in 11.3. If the test specimen cannot tolerate condensation on the surface, the dish assembly shall not be exposed to a temperature that differs by more than 5°F [3°C] from the control atmosphere to minimize the risk of condensation on the specimen. When results of water vapor transmission are expected to be less than 0.05 perm [3 ng·m<sup>-2</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>], a

dummy specimen is strongly recommended. Such a dummy specimen should be attached to an empty cup in the normal manner. The environment effects of temperature variation and buoyancy variability due to barometric pressure fluctuation can be arithmetically tared out of the weighing values. This precaution permits earlier and more reliable achievement of equilibrium conditions. Analyze the results as prescribed in 13.1.

12.4 Where water is expected to be in contact with the barrier in service, proceed as in 11.3 except place the dish in an inverted position. The dish must be sufficiently level so that water covers the inner surface of the specimen despite any distortion of the specimen due to the weight of the water. With highly permeable specimens it is especially important to locate the test dish so that air circulates over the exposed surface at the specified velocity. The test dishes may be placed on the balance in the upright position for weighing, but the period during which the wetted surface of the specimen is not covered with water must be kept to a minimum.

## 13. Calculation and Analysis of Results

13.1 The results of the rate of water vapor transmission may be determined either graphically or numerically.

13.1.1 *Dummy Specimen*—If a dummy specimen has been used to compensate for variability in test conditions, due to temperature or barometric pressure, or both, the daily recorded weights can be adjusted by calculating the weight change from initial to time of weighing. This adjustment is made by reversing the direction of the dummy's weight change, relative to its initial weight, and modifying all the appropriate specimen weight(s) recorded at this time. This permits earlier achievement of equilibrium conditions. An alternate procedure, particular for tests of long duration and more than six weighings, is to subtract the arithmetic mean slope of the rate of weight change of the dummy specimen from the arithmetic mean slope of each similar specimen to get an effective rate of weight change. These procedures are also desirable if the specimen is changing weight due to a curing process while under test.

13.1.2 *Graphic Analysis*—Plot the weight, modified by the dummy specimen when used, against elapsed time, and inscribe a curve that tends to become straight. Judgment here is required and numerous points are helpful. When a straight line adequately fits the plot of at least six properly spaced points (periodic weight changes matching, or exceeding 20 % of the multiple of 100 times the scale sensitivity), a nominally steady state is assumed, and the slope of the straight line is the rate of water vapor transmission.

13.1.3 *Numerical Analysis*—A mathematical least squares regression analysis of the weight, modified by the dummy specimen when used, as a function of time will give the rate of water vapor transmission. An uncertainty, or standard deviation of this rate, can also be calculated to define the confidence band. For very low permeability materials, this method can be used to determine the results after 30 to 60 days when using an analytical balance, with a sensitivity of  $\approx 1$  mg, even if the weight change does not meet the 100 times the sensitivity requirement of 6.3. These specimens must be clearly identified in the report.



13.2 Calculate the water vapor transmission, WVT, and permeance as follows:

### 13.2.1 Water Vapor Transmission:

$$WVT = G/tA = (G/t)/A \quad (1)$$

where:

In inch-pound units:

$G$  = weight change, grains (from the straight line),  
 $t$  = time during which  $G$  occurred, h,  
 $G/t$  = slope of the straight line, grains/h,  
 $A$  = test area (cup mouth area), ft<sup>2</sup>, and  
 $WVT$  = rate of water vapor transmission, grains/h-ft<sup>2</sup>.

In metric units:

$G$  = weight change (from the straight line), g,  
 $t$  = time, h,  
 $G/t$  = slope of the straight line, g/h,  
 $A$  = test area (cup mouth area), m<sup>2</sup>, and  
 $WVT$  = rate of water vapor transmission, g/h-m<sup>2</sup>.

### 13.2.2 Permeance:

$$\text{Permeance} = WVT/\Delta p = WVT/S(R_1 - R_2) \quad (2)$$

where:

In inch-pound units:

$\Delta p$  = vapor pressure difference, in. Hg,  
 $S$  = saturation vapor pressure at test temperature, in. Hg,  
 $R_1$  = relative humidity at the source expressed as a fraction (the test chamber for desiccant method; in the dish for water method), and  
 $R_2$  = relative humidity at the vapor sink expressed as a fraction.

In metric units:

$\Delta p$  = vapor pressure difference, mm Hg ( $1.333 \times 10^2$  Pa),  
 $S$  = saturation vapor pressure at test temperature, mm Hg ( $1.333 \times 10^2$  Pa),  
 $R_1$  = relative humidity at the source expressed as a fraction (the test chamber for desiccant method; in the dish for water method), and  
 $R_2$  = relative humidity at the vapor sink expressed as a fraction.

13.2.3 In the controlled chamber the relative humidity and temperature are the average values actually measured during the test and (unless continuously recorded) these measurements shall be made as frequently as the weight measurements. In the dish the relative humidity is nominally 0 % for the desiccant and 100 % for the water. These values are usually within 3 % relative humidity of the actual relative humidity for specimens below 4 perms [ $230 \text{ ng} \cdot \text{Pa}^{-1} \cdot \text{s}^{-1} \cdot \text{m}^{-2}$ ] when the required conditions are maintained (no more than 10 % moisture in CaCl<sub>2</sub> and no more than 1 in. [25 mm] air space above water).

13.3 The calculation of permeability is optional and can be done only when the test specimen is homogeneous (not laminated) and not less than 1/2 in. [12.5 mm] thick, calculate its average permeability as follows:

$$\text{Average permeability} = \text{Permeance} \times \text{Thickness} \quad (3)$$

13.4 *Corrections*—It is important that all applicable corrections be made to all measurements that result in permeance

value more than 2-perm [ $114 \text{ ng} \cdot \text{Pa}^{-1} \cdot \text{s}^{-1} \cdot \text{m}^{-2}$ ]. Corrections for materials with permeance value below 2-perm [ $114 \text{ ng} \cdot \text{Pa}^{-1} \cdot \text{s}^{-1} \cdot \text{m}^{-2}$ ] are insignificant and need not be done. The procedures for making various corrections, as summarized below, are found in the literature. [2,3,4,5]

13.4.1 *Buoyancy Correction*—The duration for one set of measurements can be many days or weeks. The atmospheric pressure may significantly change during such periods. If the test specimen is highly vapor resistant, the changes in mass due to vapor transport may be overshadowed by the apparent gravimetric changes observed. In such cases, all gravimetric data should be corrected to vacuum or any base line pressure. The following equation (2) can be used for buoyancy correction.

$$\frac{m_2}{m_1} = 1 + \frac{\rho_a(\rho_1 - \rho_2)}{\rho_1(\rho_2 - \rho_a)} \quad (4)$$

where:

$m_1$  = mass recorded by balance, kg,  
 $m_2$  = mass after buoyancy correction, kg,  
 $\rho_a$  = density of air, kg m<sup>-3</sup>,  
 $\rho_1$  = density of material of balance weights, kg m<sup>-3</sup>, and  
 $\rho_2$  = bulk density of test assembly, kg m<sup>-3</sup>.

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

13.4.1.2 The buoyancy correction is important (6) when measured mass changes are in the range of 0 to 100 mg.

13.4.2 *Corrections for Resistance due to Still Air and Specimen Surface*—In general, if the material is highly permeable, these corrections are more significant. With known thickness of the still air layer in the cup, the corresponding vapor resistance can be calculated using the following equation(3) for permeability.

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

where:

$\delta_a$  = permeability of still air, kg·m<sup>-1</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>,  
 $T$  = temperature, K,  
 $P$  = ambient pressure, Pa,  
 $P_o$  = standard atmospheric pressure, that is, 101325 Pa, and  
 $R_v$  = ideal gas constant for water, that is,  $461.5 \text{ J} \cdot \text{K}^{-1} \cdot \text{kg}^{-1}$ .

13.4.2.1 In the absence of any measured data, the surface resistances (that is, inside and outside surfaces of the specimen) may be approximated using Lewis' relation.(4) For cup methods that follow this standard, the total surface resistance (Hansen and Lund (5)) should be  $\approx 4 \times 10^7 \text{ Pa} \cdot \text{s} \cdot \text{m}^2 \cdot \text{kg}^{-1}$ .

13.4.3 *Edge Mask Correction*—The following equation (Joy and Wilson(1)) is to be used to correct the excess WVT effect due to edge masking:

$$\text{Percent excess WVT} = \frac{400t}{\pi S_l} \log_e \left( \frac{2}{1 + e^{-(2\pi b/t)}} \right) \quad (6)$$

where:

$t$  = specimen thickness, m,  
 $b$  = width of masked edge, m, and  
 $S_l$  = four times the test area divided by the perimeter, m.

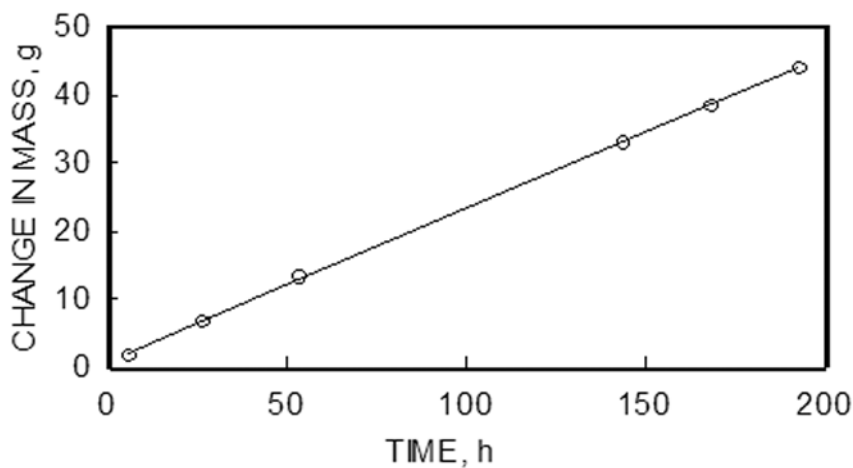


FIG. 1

13.4.3.1 If the cup assembly includes any edge masking this correction shall be made.

13.5 Metric units and conversion factor are given in Table 1.

13.6 Example (in SI unit)—In a desiccant test on a sample of medium density glass fiber insulation the following results were recorded.

Thickness of the specimen = 25.81 mm  
 Test area = 0.01642 m<sup>2</sup>  
 Mass of the test specimen = 20.44 g  
 Mass of the desiccant = 554.8 g  
 Initial mass of the test assembly = 1.257810 kg  
 Thickness of air layer in the cup = 15 mm

Elapsed Time (h)	Mass of the Test Assembly (g)	Change in Mass (g)	Chamber Temperature (°C)	Chamber RH (%)	Barometric Pressure mm Hg (kPa)
0.000	1257.810	0.000	22.83	52.60	744.7 (99.27)
6.067	1259.469	1.659	22.84	52.6	741.11 (98.79)
26.633	1264.609	6.799	22.78	52.2	744.41 (99.23)
53.150	1271.062	13.252	22.82	52.1	743.21 (99.07)
143.767	1290.773	32.963	22.74	52.2	757.69 (101.00)
168.283	1296.389	38.579	22.78	52.1	749.81 (99.95)
192.883	1301.953	44.143	22.78	52.1	758.44 (101.10)

13.6.1 Buoyancy Correction—As mentioned in 13.4.1, the buoyancy effect will be insignificant for this set of readings as recorded changes of mass are all above 100 mg. However, for example, the corrected mass of the test assembly weight 1257.810 g (1<sup>st</sup> reading) can be calculated using Eq 4.

$$\begin{aligned}
 m_1 &= \text{mass recorded by balance, kg} = 1257.810 \times 10^{-3} \text{ kg} \\
 P &= \text{Barometric pressure, Pa} = 99.27 \times 10^3 \text{ Pa} \\
 R &= \text{Gas constant for dry air} = 287.055 \text{ J / (kg·K)} \\
 T &= \text{Chamber temperature} = 22.83 + 273.15 = 295.98 \text{ K} \\
 \rho_a &= \text{density of air, kg m}^{-3} = P / (RT) = 1.1684 \text{ kg m}^{-3} \\
 \rho_1 &= \text{density of material of balance weights, kg m}^{-3} = 8000 \text{ kg m}^{-3} \\
 h_1 &= \text{height of the test assembly, m} = 44.7 \times 10^{-3} \text{ m} \\
 d_1 &= \text{diameter of the test assembly, m} = 168.0 \times 10^{-3} \text{ m} \\
 \rho_2 &= \text{bulk density of test assembly, kg m}^{-3} \\
 &= \frac{4 \times m_1}{\pi \times d_1^2 \times h_1} = 1269.4 \text{ kg m}^{-3} \\
 m_2 &= \text{mass after buoyancy correction} = 1258.78 \times 10^{-3} \text{ kg}
 \end{aligned}$$

13.6.2 A graphic analysis of the data, according to 13.1.2 is shown in Fig. 1.

13.6.3 A linear least-squares analysis of the data according to 13.1.3 gives the slope of the straight line as  $0.225 \pm 0.002 \text{ g·h}^{-1}$ , with a linear regression coefficient > 0.998.

$$\begin{aligned}
 \text{WVT} &= 0.225 \text{ g·h}^{-1} / 0.01642 \text{ m}^2 \\
 &= 19.595 \text{ grains·h}^{-1}\text{·ft}^{-2} (\approx 3.81 \times 10^6 \text{ ng·m}^{-2}\text{·s}^{-1}) \\
 S &= 2775.6 \text{ Pa} \\
 R_1 &= 0.523 \\
 R_2 &= 0 \\
 \text{Permeance} &= 3.81 \times 10^6 \text{ ng·m}^{-2}\text{·s}^{-1} / (2775.6 \text{ Pa} \times 0.523) \\
 &= 2630 \text{ ng·m}^{-2}\text{·s}^{-1}\text{·Pa}^{-1}
 \end{aligned}$$

13.6.4 Corrections for Resistance due to Still Air and Specimen Surface:

$$\begin{aligned}
 \text{Permeability of still air layer (Eq 5)} \\
 \delta_a &= \frac{2.306 \times 10^{-5} \times 101325}{461.5 \times (22.79 + 273.15) \times 99860} \left( \frac{22.79 + 273.15}{273.15} \right)^{1.81} \\
 &= 198 \text{ ng·m}^{-1}\text{·s}^{-1}\text{·Pa}^{-1} \\
 \text{Permeance of 15 mm still air layer} \\
 &= (198) / (0.015) \text{ ng·m}^{-2}\text{·s}^{-1}\text{·Pa}^{-1} \\
 &= 13200 \text{ ng·m}^{-2}\text{·s}^{-1}\text{·Pa}^{-1} \\
 \text{Hence, the 15 mm air layer offers a vapor resistance} \\
 &= 1 / (13200) \text{ m}^2\text{·s·Pa·ng}^{-1} \approx 7.6 \times 10^7 \text{ m}^2\text{·s·Pa·kg}^{-1} \\
 \text{Surface resistances (see 13.4.2)} \\
 &\approx 4.0 \times 10^7 \text{ m}^2\text{·s·Pa·kg}^{-1} \\
 \text{Total corrections for resistance due to still air and specimen surface} \\
 &= (7.6 \times 10^7 + 4.0 \times 10^7) \text{ m}^2\text{·s·Pa·kg}^{-1}
 \end{aligned}$$

13.6.5 Edge Mask Correction—The test assembly used does not include any edge masking. However, for example, if it includes an edge mask of width 5 mm then the following correction is to be made (see 13.4.3).

$$\begin{aligned}
 t &= \text{specimen thickness, m} = 25.81 \times 10^{-3} \text{ m} \\
 b &= \text{width of masked edge, m} = 5 \times 10^{-3} \text{ m} \\
 \text{Test area} &= 0.01642 \text{ m}^2 \\
 \text{Perimeter} &= 0.4541 \text{ m} \\
 S_1 &= \text{four times the test area divided by the perimeter} \\
 &= \frac{4 \times 0.01642}{0.4541} = 0.1446 \text{ m} \\
 \text{Percent excess WVT} \\
 &= \frac{400 \times 25.81 \times 10^{-3}}{\pi \times 0.1446} \log_e \left( \frac{2}{1 + e^{-(2\pi \times 5 \times 10^{-3}) / (25.81 \times 10^{-3})}} \right) \\
 &= 9.86 \%
 \end{aligned}$$

13.6.6 The applicable corrections required for the analysis of the test results in this case are due to resistance of still air and specimen surface.

**TABLE 2 Results on Precision from Interlaboratory Testing**

For Desiccant Method at 23°C			Repeatability			Reproducibility		
Material	Thickness (mm) <sup>A</sup>	Mean Permeance (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>	s (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>	CV (%)	LSD (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>	s (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>	CV (%)	LSD (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>
A	0.0254	34.7	0.95	2.7	2.7	5.6	16.2	15.9
B	0.1397	0.74	0.16	21.7	0.46	0.31	42.6	0.92
C	12.7	3.51	0.25	7.2	0.69	1.06	30.2	2.8
D	25.4	44.8	1.5	3.3	4.2	3.5	7.8	10.0
E	0.3556	2.64	0.13	5.0	0.40	0.31	11.7	0.86

For Water Method at 23°C			Repeatability			Reproducibility		
Material	Thickness (mm) <sup>A</sup>	Mean Permeance (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>	s (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>	CV (%)	LSD (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>	s (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>	CV (%)	LSD (ng·m <sup>-2</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>B</sup>
A	0.0254	40.91	0.77	1.9	2.2	8.9	21.8	25.2
B	0.1397	0.90	0.13	14.0	0.35	0.12	13.4	0.34
C	12.7	5.55	0.31	5.7	0.92	1.1	20.1	3.1
D	25.4	59.5	1.1	1.8	3.1	12.4	20.9	35.5
E	0.3556	3.40	0.19	5.7	0.57	0.47	13.8	1.3

<sup>A</sup> 1 in. = 25.4 mm

<sup>B</sup> 1 perm (inch-pound) = 57.2 ng·m<sup>-2</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>

Legend:

s = standard deviation

CV = percent coefficient of variation ( $s \times 100 / \text{Mean}$ )

LSD = least significant difference between two individual test results based on a 95 % confidence level =  $2\sqrt{2s}$

NOTE 1—Material B was Teflon<sup>5</sup> PTFE fluorocarbon resin brand of tetrafluoroethylene. It was extremely difficult to provide a seal to this sample, which accounts for the poor repeatability.

Water vapor resistance of the test specimen + corrections

$$= 1 / \text{Permeance} = (1 / 2630) \text{ m}^2 \cdot \text{s} \cdot \text{Pa} \cdot \text{ng}^{-1}$$

$$= 3.80 \times 10^8 \text{ m}^2 \cdot \text{s} \cdot \text{Pa} \cdot \text{kg}^{-1}$$

The water vapor resistance of the test specimen

$$= (3.80 \times 10^8 - (7.6 \times 10^7 + 4.0 \times 10^7)) \text{ m}^2 \cdot \text{s} \cdot \text{Pa} \cdot \text{kg}^{-1}$$

$$= 2.64 \times 10^8 \text{ m}^2 \cdot \text{s} \cdot \text{Pa} \cdot \text{kg}^{-1}$$

Permeance of the test specimen

$$= 1 / (2.64 \times 10^8 \text{ m}^2 \cdot \text{s} \cdot \text{Pa} \cdot \text{kg}^{-1})$$

$$= 3.79 \times 10^{-9} \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$$

$$= 3790 \text{ ng} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$$

Permeability

$$= 3790 \text{ ng} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1} \times 0.02581 \text{ m}$$

$$= 97.8 \text{ ng} \cdot \text{m}^{-1} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$$

## 14. Report

14.1 The report shall include the following:

14.1.1 Identification of the material tested, including product thickness for homogeneous materials (not laminated) greater than 1/2 in.,

14.1.2 Test method used (desiccant or water),

14.1.3 Test temperature,

14.1.4 Relative humidity in the test chamber,

14.1.5 Permeance of each specimen in perms (to two significant figures),

14.1.6 The side of each specimen on which the higher vapor pressure was applied. (The sides shall be distinguished as “side A” and “side B” when there is no obvious difference between them. When there is an obvious difference, this difference shall also be stated, such as “side A waxed” and “side B unwaxed.”),

14.1.7 The average permeance of all specimens tested in each position,

14.1.8 The permeability of each specimen (as limited by 13.3), and the average permeability of all specimens tested,

14.1.9 Include a portion of the plot indicating the section of the curve used to calculate permeability, and

14.1.10 State design of cup and type or composition of sealant.

## 15. Precision and Bias

15.1 *Precision*—Table 2 is based on interlaboratory tests conducted in 1988 and 1991.<sup>5</sup> In 1988 four materials (A, B, C, D) were tested using the desiccant method and the water method in triplicate. Fifteen laboratories contributed data, with full results secured from four laboratories. In 1991 ten laboratories contributed data for material E, using triplicate specimens, again using both the desiccant method and the water method. Tables 3 and 4 are based on another interlaboratory test conducted in 1995–96.<sup>(7)</sup> One material at a nominal thickness of 1 in. (25 mm) was tested by ten participating laboratories. Results from only nine laboratories were used in the analyses because of the presence of severe outliers (see Practice E691) in the observation of tenth laboratory.

15.1.1 Test results were analyzed using Practice E691.

15.2 Additional precision data and analysis for this test method is based on an interlaboratory study (#512) conducted in 2010. Six laboratories participated in this study, analyzing four different extremely low permeance materials. Procedure A, desiccant method at 73°F/50% RH, was used. Each “test result” reported represents an individual determination, and all participants reported three replicate test results for every material. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. C16-1040.<sup>6</sup>

15.2.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the “r” value for that material; “r” is the

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C16-1014.

<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C16-1040. Contact ASTM Customer Service at service@astm.org.

**TABLE 3 Results on Precision from Interlaboratory Testing—Dry Cup Measurements on Expanded Polystyrene**

Lab	Permeability (ng·m <sup>-1</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>A</sup>			$\bar{x}$	s	d	h	k
	Spec #1	Spec #2	Spec #3					
1	2.54	2.46	2.21	2.40	1.72E-01	-7.01E-01	-1.50	1.06
2	2.65	2.87	2.68	2.73	1.19E-01	-3.71E-01	-0.79	0.73
3	3.79	3.49	3.65	3.64	1.50E-01	5.39E-01	1.15	0.92
4	2.77	2.73	2.69	2.73	4.00E-02	-3.74E-01	-0.80	0.25
5	2.67	2.66	2.79	2.71	7.23E-02	-3.98E-01	-0.85	0.44
6	3.26	3.38	3.29	3.31	6.24E-02	2.06E-01	0.44	0.38
7	3.05	3.72	3.33	3.37	3.37E-01	2.62E-01	0.56	2.07
8	3.76	3.53	3.87	3.72	1.73E-01	6.16E-01	1.31	1.07
9	3.24	3.48	3.26	3.33	1.33E-01	2.22E-01	0.47	0.82
				$\bar{\bar{x}}$	$s_r$	$s_{\bar{x}}$	$s_R$	
				3.10	1.63E-01	4.69E-01	4.87E-01	

<sup>A</sup> 1 perm in. = 1.45 (ng·m<sup>-1</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>)

NOTE 1—The average of the cell averages gives the permeability for the round robin material, according to the dry cup measurements, as 3.10 ng·m<sup>-1</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>.

NOTE 2—The repeatability standard deviation is  $1.6 \times 10^{-1}$  ng·m<sup>-1</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>.

NOTE 3—The reproducibility standard deviation is  $4.9 \times 10^{-1}$  ng·m<sup>-1</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>.

**TABLE 4 Results on Precision from Interlaboratory Testing—Wet Cup Measurements on expanded polystyrene**

Lab	Permeability (ng·m <sup>-1</sup> ·s <sup>-1</sup> ·Pa <sup>-1</sup> ) <sup>A</sup>			$\bar{x}$	s	d	h	k
	Spec #1	Spec #2	Spec #3					
1	2.90	3.14	2.94	2.99	1.29E-01	-3.58E-01	-0.94	0.77
2	3.50	3.46	3.52	3.49	3.06E-02	1.43E-01	0.37	0.18
3	4.23	3.76	3.65	3.88	3.08E-01	5.29E-01	1.39	1.84
5	3.32	3.29	2.97	3.19	1.94E-01	-1.58E-01	-0.41	1.16
6	2.61	2.82	2.80	2.74	1.16E-01	-6.08E-01	-1.59	0.69
7	3.53	3.18	3.41	3.37	1.77E-01	1.92E-02	0.05	1.06
8	3.30	3.42	3.29	3.34	7.23E-02	-1.42E-02	-0.04	0.43
9	3.75	3.97	3.67	3.80	1.55E-01	4.46E-01	1.17	0.93
				$\bar{\bar{x}}$	$s_r$	$s_{\bar{x}}$	$s_R$	
				3.35	1.67E-01	3.82E-01	4.06E-01	

<sup>A</sup> 1 perm in. = 1.45 ng·m<sup>-1</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>

Legend:

$\bar{x}$  = Cell average or the average from one laboratory

s = Cell standard deviation, or the standard deviation for one laboratory

$\bar{\bar{x}}$  = Average of the Cell averages

d = Cell deviation or the difference ( $\bar{x} - \bar{\bar{x}}$ )

$s_r$  = Repeatability standard deviation (within a laboratory)

$s_R$  = Reproducibility standard deviation (between the laboratories)

h = the between-laboratory consistency statistic

k = the within-laboratory consistency statistic

NOTE 1—The average of the cell averages gives the permeability for the round robin material, according to the wet cup measurements, as 3.35 ng·m<sup>-1</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>.

NOTE 2—The repeatability standard deviation is  $1.7 \times 10^{-1}$  ng·m<sup>-1</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>.

NOTE 3—The reproducibility standard deviation is  $4.1 \times 10^{-1}$  ng·m<sup>-1</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>.

interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

15.2.1.1 Repeatability limits are listed in Table 5 below.

15.2.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the “R” value for that material; “R” is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

15.2.2.1 Reproducibility limits are listed in Table 5 below.

15.2.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

15.2.4 Any judgment in accordance with statements 9.1.1 and 9.1.2 would have an approximate 95% probability of being correct.

15.3 The precision statement was determined through statistical examination of 72 test results, submitted by six laboratories, for four materials. The four materials were described as:

Material A: 6 mil high barrier PVdC-based film

Material B: 10 mil high barrier PVdC-based film

Material C: 15 mil high barrier HDPE-based film

Material D: PET film/1 mil aluminum foil/PET film lamination

**TABLE 5 Water Vapor Transmission (perms) ILS #512**

Material	Average <sup>A</sup>	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	$s_r$	$s_R$	$r$	$R$
A	0.00877	0.00166	0.00336	0.00465	0.00940
B	0.00843	0.01067	0.01067	0.02988	0.02988
C	0.02028	0.01698	0.01698	0.04756	0.04756
D	0.00567	0.00938	0.00967	0.02626	0.02706

<sup>A</sup> The average of the laboratories' calculated averages

15.4 To judge the equivalency of two test results, it is recommended to choose the material that is closest in characteristics to the test material.

15.5 Using information from this ILS, Appendix section X3 discusses the testing of extremely low permeance materials.

## 16. Keywords

16.1 permeability; plastics (general); plastic sheet and film; sheet material; thermal-insulating materials; thermal insulation permeability films; water vapor transmission (WVT)

## APPENDIXES

### (Nonmandatory Information)

#### X1. STANDARD TEST CONDITIONS

X1.1 Standard test conditions that have been useful are:

X1.1.1 *Procedure A*—Desiccant Method at 73.4°F [23°C].

X1.1.2 *Procedure B*—Water Method at 73.4°F [23°C].

X1.1.3 *Procedure BW*—Inverted Water Method at 73.4°F [23°C].

X1.1.4 *Procedure C*—Desiccant Method at 90°F [32.2°C].

X1.1.5 *Procedure D*—Water Method at 90°F [32.2°C].

X1.1.6 *Procedure E*—Desiccant Method at 100°F [37.8°C].

#### X2. CUP DESIGN AND SEALING METHODS

X2.1 An ideal sealing material has the following properties:

X2.1.1 Impermeability to water in either vapor or liquid form.

X2.1.2 No gain or loss of weight from or to the test chamber (evaporation, oxidation, hygroscopicity, and water solubility being undesirable).

X2.1.3 Good adhesion to any specimen and to the dish (even when wet).

X2.1.4 Complete conformity to a rough surface.

X2.1.5 Compatibility with the specimen and no excessive penetration into it.

X2.1.6 Strength or pliability (or both).

X2.1.7 Easy handleability (including desirable viscosity and thermal of molten sealant).

X2.1.8 Satisfactory sealants possess these properties in varying degrees and the choice is a compromise, with more tolerance in items at the beginning of this list for the sake of those at the latter part of the list when the requirements of 7.2 are met. Molten asphalt or wax is required for permeance tests below 4 perms [240 ng·m<sup>-2</sup>·s<sup>-1</sup>·Pa<sup>-1</sup>]. Tests to determine sealant behavior should include:

X2.1.8.1 An impervious specimen (metal) normally sealed to the dish and so tested, and

X2.1.8.2 The seal normally assembled to an empty dish with no specimen and so tested.

X2.2 The following materials are recommended for general use when the test specimen will not be affected by the temperature of the sealant:

X2.2.1 Asphalt, 180 to 200°F [82 to 93°C] softening point, meeting the requirements of Specification D449, Type C. Apply by pouring.

X2.2.2 Beeswax and rosin (equal weights). A temperature of 275°F [135°C] is desirable for brush application. Pour at lower temperature.

X2.2.3 Microcrystalline wax<sup>7</sup> (60 %), mixed with refined crystalline paraffin wax (40 %).

X2.3 The materials listed in X2.3.1 are recommended for particular uses such as those shown in Fig. X2.1. The suggested procedure described in X2.3.2 applies to an 11<sup>3</sup>/<sub>8</sub>-in. [289-mm]

<sup>7</sup> The sole source of supply of the microcrystalline wax known to the committee at this time is E. I. DuPont de Nemours & Co., Inc., Polymer Products Dept., Wilmington, DE 19898. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.



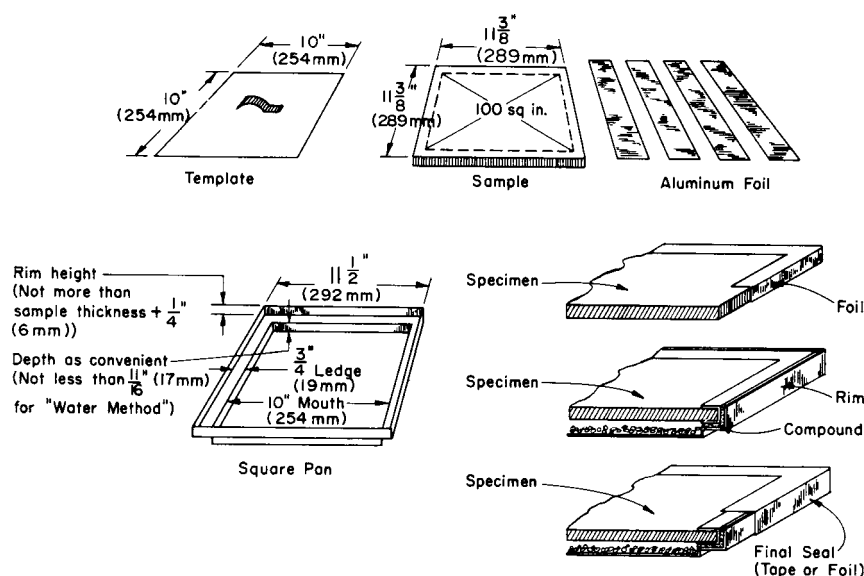


FIG. X2.1 Apparatus for Water Vapor Transmission Tests of Large Thick Specimens

square specimen if its permeance exceeds 4 perms [ $240 \text{ ng} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ ] (limited by evaporation of sealants).

#### X2.3.1 Materials:

X2.3.1.1 Aluminum foil, 0.005 in. [0.125 mm] minimum thickness.

X2.3.1.2 Tape, meeting the requirements of Specification D2301, vinyl chloride plastic pressure-sensitive, electrical insulating tape.

X2.3.1.3 Cement, contact bond, preferably rubber base.

#### X2.3.2 Procedure:

X2.3.2.1 *Step 1*—Seal aluminum foil around edges of specimen, leaving a 100-in.<sup>2</sup> [0.0654-m<sup>2</sup>] exposed test area on each side. Use contact bond cement as directed by the manufacturer.

X2.3.2.2 *Step 2*—Spread sealant on inside of rim and ledge. Place desiccant (dry), or water and surge control material (wet) in pan. Press specimen in place. Avoid squeezing compound into the test area.

X2.3.2.3 *Step 3*—Coat outside of rim and bottom of ledge with contact bond cement, and place foil strips from edge of template, around rim, and bottom of ledge.

X2.4 A method of using hot asphalt, as applied to a 10-in. [254-mm] square-mouth dish with ledge and rim, is as follows:

#### X2.4.1 Apparatus:

X2.4.1.1 *Template*—A square frame of brass or steel,  $\frac{3}{16}$  in. [5 mm] thick and  $\frac{3}{4}$  in. [19 mm] deep. The  $\frac{3}{16}$ -in. [5-mm] thickness is tapered to zero at the bottom of the frame where it will touch the test specimen and maintain a 10-in. [254-mm] square test area.

X2.4.1.2 *Sealant*—Asphalt (see X2.3.1 used at the proper pouring consistency of 375 to 450°F [179 to 232°C]).

X2.4.1.3 *Melting Pot*, for the asphalt, electrically heated, with one dimension greater than 11  $\frac{3}{8}$  in. [289 mm].

X2.4.1.4 *Small Ladle*, for pouring.

X2.4.2 *Procedure*—Mark the 11  $\frac{3}{8}$ -in. [289-mm] square specimen with a line at an equal distance from each edge, so

that the area enclosed by the lines is as nearly as possible a 10-in. [254-mm] square. The template may be used for marking. Dip each edge of the specimen in molten asphalt up to the line, so that the test area is defined and all edges are coated with a heavy layer of asphalt. Place the specimen over the pan containing water or desiccant. Lightly oil the template or coat with petroleum jelly on its outer side, and place on the specimen. Pour molten asphalt into the space between the template and the rim of the pan. After the asphalt has cooled for a few minutes, the template should be easily removable.

X2.5 Hot wax may be applied like asphalt. It may also be applied (freely) with a small brush. Its lower working temperature may be advantageous when a specimen contains moisture.

X2.6 Several designs for dishes with supporting rings and flanges are shown in Fig. X2.2. Various modifications of these designs may be made provided that the principle of prevention of edge leakage by means of a complete seal is retained. The dishes may be constructed of any rigid, impermeable, corrosion-resistant material, provided that they can be accommodated on the available analytical balance. A lightweight metal, such as aluminum or one of its alloys, is generally used for larger-size dishes. In some cases when an aluminum dish is employed and moisture is allowed to condense on its surface, there may be appreciable oxidation of the aluminum with a resulting gain in weight. Any gain in weight will ordinarily depend on the previous history of the dish and the cleanness of the surface. An empty dish carried through the test procedure as a control will help to determine whether any error may be expected from this cause. When aluminum dishes are used for the water methods, a pressure may develop inside the assembly during a test due to corrosion. This can cause seal failure or otherwise affect the result. Where this is a problem, it can be overcome by providing inside the dish a protective coating of baked-on epoxy resin or similar material. Dishes with flanges or rings that project from the inner walls of the dish are to be avoided, as such projections influence the diffusion of the water vapor. The depth of the dish for the water procedures is

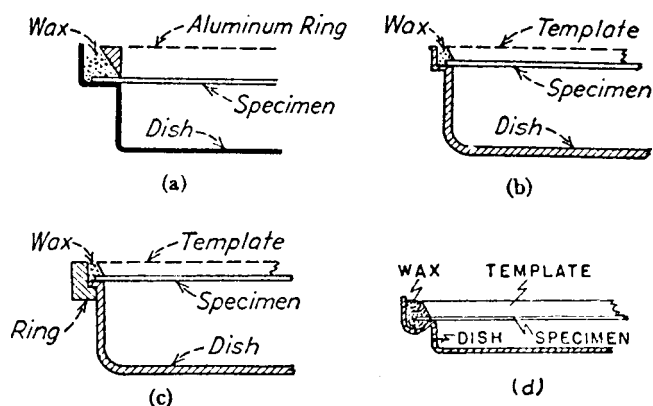


FIG. X2.2 Several Types of Dishes for Water Vapor Transmission Tests of Materials in Sheet Form

such that there is a  $0.80 \pm 0.20$ -in. [ $20 \pm 5$ -mm] distance between the water surface and the under surface of the specimen, with a water depth of about 0.20 in. [5 mm].

X2.6.1 For the desiccant-in-dish procedures, the dishes need not be as deep as those required for the water-in-dish procedures. The desiccant is within  $\frac{1}{4}$  in. [6 mm] of the under surface, and a minimum depth of only  $\frac{1}{2}$  in. [12 mm] of desiccant is required.

X2.6.2 The dishes shown in Fig. X2.2 require a molten seal.

X2.6.3 A template such as is shown in Fig. X2.3 is usually used for defining the test area and effecting the wax seal. It consists of a circular metal dish  $\frac{1}{8}$  in. [3.18 mm] or more in thickness with the edge beveled to an angle of about  $45^\circ$ . The diameter of the bottom (smaller) face of the template is approximately equal to, but not greater than, the diameter of the effective opening of the dish in contact with the specimen. Small guides may be attached to the template to center it automatically on the test specimen. A small hole through the template to admit air, and petrolatum applied to the beveled edge of the template facilitate its removal after sealing the test specimen to the dish. In use, the template is placed over the test

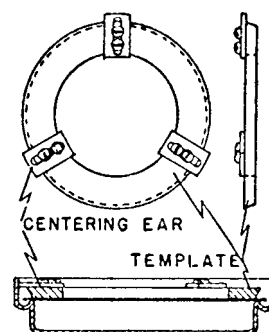


FIG. X2.3 Template Suitable for Use in Making the Wax Seals on Test Dishes

specimen and when it is carefully centered with the dish opening, molten wax is flowed into the annular space surrounding the beveled edge of the template. As soon as the wax has solidified, the template is removed from the sheet with a twisting motion. The outside flange of the dish should be high enough to extend over the top of the specimen, thus allowing the wax to completely envelop the edge.

X2.6.4 Gasketed types of seals are also in use on appropriately designed dishes. These simplify the mounting of the specimen, but must be used with caution, since the possibility of edge leakage is greater with gasketed seals than with wax seals. Gasketed seals are not permitted for the measurement of permeance less than 4 perms [ $240 \text{ ng} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ ]. As a further precaution when gasketed seals are used instead of preferred sealants, a blank test run is suggested using glass or metal as a dummy specimen.

X2.6.5 A suitable weighing cover consists of a circular disk of aluminum  $\frac{1}{32}$  to  $\frac{3}{32}$  in. [0.8 to 2.4 mm] in thickness provided with a suitable knob in the center for lifting. The cover fits over the test specimen when assembled and makes contact with the inside beveled surface of the wax seal at, or just above, the plane of the specimen. The cover is free of sharp edges that might remove the wax and is numbered or otherwise identified to facilitate its exclusive use with the same dish.

### X3. PROCEDURE TO CALCULATE DEPENDENCY OF WATER VAPOR TRANSMISSION RATE ON RELATIVE HUMIDITY

X3.1 The dependency of the water vapor transmission (WVT) rate of materials on relative humidity (RH) can be determined using a combination of desiccant and water method (8).

#### X3.2 Procedure

X3.2.1 Dry cup tests with *desiccant method* test set up but also with additional chamber RH levels other than 50% are carried out. Three chamber RH levels: 50%, 70%, and 90% shall be selected.

X3.2.2 Wet cup measurements with water method test set up but at two chamber RH levels are to be carried out. Two chamber RH levels, 70% and 90% shall be selected.

#### X3.3 Data Analysis

X3.3.1 From the slope of the time versus mass change data plot, for each chamber RH levels, the rate of WVT for the corresponding test specimen at a specific chamber RH level is determined according to 13.2.1.

X3.3.2 The WVT rate is plotted against the chamber RH. This results in two separate but intersecting plots.

X3.3.2.1 If the plots are linear and intersect at 50% chamber RH, it is concluded that the material under consideration is nonhygroscopic and the rate of WVT does not depend on the local chamber RH.

X3.3.2.2 For the hygroscopic material the intersection of the plots will be at a chamber RH greater than 50% and water method may yield a nonlinear dependency.

X3.3.3 For hygroscopic materials the sum of the rate of WVT from dry and wet cup measurements at the 90% chamber RH represents theoretically the dry cup measurements for WVT rate done at 100% chamber RH.

X3.3.4 Similarly for hygroscopic materials the sum of the rate of WVT from dry and wet cup measurements at the 70% chamber RH represents theoretically the dry cup measurements for WVT rate done at 100% chamber RH. If this calculated value of WVT rate at 100% chamber RH differs from the same calculated in centilitres X3.3.3 by more than 10% then the whole test procedure should be repeated.

X3.3.5 The calculated dry cup WVT rate at 100% chamber RH, as shown above, is combined with the directly measured dry cup measurements data at 50%, 70% and 90% chamber RH

to generate a set of WVT rate values spanned over the entire range of chamber RH (that is, 0 to 100%). These values of WVT rate when plotted against the corresponding chamber RH would define the dependency of WVT rate on RH.

X3.3.6 The algebraic expression of the best-fitted curve that passes through the origin from the WVT rate versus chamber RH is to be used to determine the derivative of the plot at any given local RH.

X3.3.7 The water vapor permeance of the material at a specific chamber RH is derived from the following expression.

$$\text{Water Vapor Permeance (WVP)} = \quad (X3.1)$$

$$\left\{ \frac{(\text{Magnitude of derivative}) \times 100}{\text{Saturation water vapor pressure at } 73.4^{\circ}\text{F } (23^{\circ}\text{C})} \right\}$$

X3.3.8 All normal required corrections (13.4) are applicable to X3.3.7.

#### X4. TESTING OF EXTREMELY LOW PERMEANCE MATERIALS

X4.1 In 2010-2011 an inter-laboratory study using Procedure A was conducted using thin, extremely low permeance materials as the test specimens. The statistics from this study related to precision of the method are covered in Section 15 of these test methods.

X4.2 In addition to developing a precision statement, a number of other objectives was targeted in undertaking this round robin. Those objectives and a discussion of the findings are discussed herein:

X4.3 Provide experience testing extremely low and “zero perm” materials.

X4.3.1 Many labs that conduct the E96/E96M tests do not have occasion to test such materials. The materials tested in the study ranged from anticipated values of about 0.015 perm down to zero perm. All materials were 0.015 in. (0.38 mm) thick or less. Circumstances were such, however, that the final group of participating labs in fact did have experience testing in this range. As such, input from inexperienced labs was not available.

X4.4 Determine what difficulties are encountered in testing at these levels.

X4.4.1 With one exception, no notable difficulties were reported by the labs. One lab was consistently obtaining loss of weight during the tests. An assignable cause was not determined, and it was decided not to use their results. Otherwise, there was no feedback on problems with the test.

X4.4.2 The occurrence of outliers indicates problems that were undetected or uncorrected by the operators, or both. The outliers are always high results. Absent the presence of obvious defects in the test specimens, which would be visible under normal lighting or over a light box, the cause for outliers is invariably inadequate sealing to the dish. It would appear that operators were not recognizing outliers appearing early in the

test, or were not taking action to correct faulty seals or otherwise determine a cause.

X4.4.3 The types of materials tested can be expected to be very consistent. One exception could be foil or foil laminations, which can contain pinholes of a size or number that can produce apparently outlying results. These will be detectable over a light box. The foil lamination samples in this study were pre-screened for presence of pinholes, and only pinhole-free specimens were used.

X4.4.4 The primary problem encountered in testing extremely low permeance materials then would appear to be inadequate or failing seals. It is critical that operators monitor data early on for apparent outliers. When one is suspected, whether or not a breach in the seal is visible in the test dish, the seal should be “re-flowed”, the specimen taken out of the test, or a replacement replicate started.

X4.4.5 It is strongly recommended that a program be set up to provide either water vapor transmission rate or permeance, and correlation coefficient computation, real time at each weighing. This way outliers can be spotted immediately and checked for cause.

##### X4.5 Application of correlation coefficient:

X4.5.1 The correlation coefficient indicates if a strong linear relationship in the coordinate data points exists.

X4.5.2 Very high correlation indicates a very straight line slope for the weight gain per unit time, which in turn can be used as an indication of steady state.

X4.5.3 High correlation is not necessarily expected when testing materials of extremely low permeance, since the slope of the weight gain per unit time is approaching zero, and any variation in the test conditions has a greater impact on individual weighings. Zero or near-zero permeance tests may never show good correlation.



#### X4.6 Time required to reach steady state:

X4.6.1 It is generally thought that extremely low permeance materials require many weeks or months to reach a true steady state and to provide a reliable result.

X4.6.2 A graph of perm vs. time at steady state should show a flat line.

X4.6.3 Using the data from one lab that obtained the most consistent expected results, the approximately first one third of the test duration (18 days) showed perm results that indicated a condition that was not steady-state.

X4.6.4 The second third (19-36 days) graph showed a much straighter line, but one not totally flat.

X4.6.5 The last third (37-54 days) showed the straightest you line of perm results; given the range of the data, steady state is well indicated.

X4.6.6 While the above findings would indicate that two months or more may be needed to reach steady state, the change in results after the first two or three weeks was very small, less than would impact a result rounded to two significant figures. The purpose of the test might dictate the duration required; that is, screening or QC tests might be run for shorter durations than R&D tests where a high degree of accuracy is desired, which might be run for more than two months .

X4.6.7 It is known that thicker materials and moisture-retaining materials need upwards of two months or longer to reach steady-state. Evidence shows that very thin materials such as those tested in this ILS, even if of extremely low permeance, may not need such a long test duration to reach steady state.

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