## BUILT HERITAGE EVALUATION

## **Manual Using Simple Test Methods**

## **EDITED BY**

A. ELENA CHAROLA JORGE OTERO PAULA T. DEPRIEST ROBERT J. KOESTLER



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A Smithsonian Contribution to Knowledge



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## **PREFACE**

The idea for this manual came from our interactions with colleagues in Latin America, a region where there are few resources for preserving architectural heritage and where conservators may not receive practical, hands-on experience during their training. In the hopes of assisting our colleagues in similarly under-resourced regions around the globe, we have created this publication: a manual that brings together simple tests for use in evaluating the state of conservation of architectural heritage. Conservators, architects, archaeologists, and other heritage professionals will find these methods useful for examining and evaluating the condition of historic building materials and for choosing conservation and restoration products that are appropriate and not harmful and that will not prevent subsequent treatments.

One of the problematic issues for built heritage is that regular maintenance is often not "regular" but rather haphazard. We may think of historic structures as resilient and perennial because they have survived so long. However, these structures may develop urgent problems at any time: gutters that fail, mortars that crumble, stone blocks that crack. The main issue is that historic landmarks become so familiar that they are no longer "seen" even by the people tasked with their protection. Professional conservators need training to observe small changes, even when they are gradual, before they reach a breaking point and become catastrophic. At that point, the solution to the problem is often far more costly and intrusive than if it had been detected and addressed earlier. The tests presented here are important tools for "monitoring" of the building and detecting of problems early.

For example, consider a historic brick masonry building. Over the years, the lime mortar in the joints eroded and is patched with a cement mortar. The latter, being a less porous material, increased the water content in the bricks and accelerated their deterioration, especially if they were subjected to winter freeze-thaw cycles. This deterioration of the brick could have been slowed or avoided if the mortar had been replaced with a formulation similar to the original, which was compatible with the brick. The switch to a cement mortar triggered the deterioration. The problem now is far more serious than the original erosion of the lime mortar.

The aim of this manual is to provide simple and useful test methods for conservators who do not have ready access to laboratories specialized in the analysis and evaluation of building materials and their deterioration or in the evaluation of conservation treatments. The tests described will provide for the preliminary evaluation of a material, its condition, and its performance, especially when exposed to the most significant deterioration factor for our built environment—water. In many cases in which there is no severe damage,

results from these tests should be enough to develop a long-term maintenance plan that is fundamental for the protection of a building or monument.

We hope that the present manual will prove useful to those who are responsible for the first assessment of the problems in the conservation and preservation of built heritage.

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All photographs for the manual were taken by Jorge Otero, and all graphs were created by either Jorge Otero or A. Elena Charola.

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## HOW TO USE THIS MANUAL

## A. ELENA CHAROLA

The objective of this manual is to bring together simple tests for under-resourced professionals, providing methods to characterize the condition of historic building materials and describing tests to use for evaluation of conservation materials. The tests were selected based on their simplicity and widespread availability, especially for countries where few institutions deal with the conservation of built heritage and scarce laboratories provide analyses necessary to evaluate the condition of an important building or monument.

This short manual describes different tools and tests that can be routinely used for characterizing the building or monument materials, such as stone, brick, mortar or adobe. These methods are simple and require a minimum of supplies and instrumentation. Some can be carried out in the field; others need a basic laboratory. There are several options for portable microscopes depending on the type of material to be evaluated: digital microscopes; simple optical microscopes; and the recently developed and low-cost origami-based paper microscope ("Foldscope"). The presence of salts can be evaluated directly on a building or monument by using simple paper tests strips that detect different cations and anions. The strips can provide a preliminary concentration for the anion or cation to determine if there is a problem and to detect the source of the contamination. More detailed quantification would require laboratory facilities.

The remaining chapters include some standard tests. For example, the Scotch Tape test (ASTM 4214-97, 1997), developed at the International Centre for the Study of the Preservation and Restoration of Cultural Property (ICCROM), evaluates the surface of a material to determine whether it is sound or "sanding." A strip of Scotch Tape is adhered to the surface and then pulled off and weighed. Its increase in weight corresponds to the amount of surface material removed and indicates the degree of surface deterioration. It is very useful for evaluating whether treatment has improved the condition of the surface. The RILEM tube water absorption test (RILEM Test Method No. II.4, 2015) is a standard test for evaluating in situ water absorption. The water vapor transmission test (following ASTM E 96/E 96M, 2005) has been simplified significantly. This test evaluates how fast water vapor passes through a stone sample and indicates the stone's property as a moisture barrier, which is important especially when a surface treatment, such as paint or a water repellent, is considered. This test can be used to evaluate the effectiveness of a coating treatment by comparing the before and after results.

Last but not least, three separate standard tests are applied sequentially: water absorption coefficient (RILEM Test No. II-6, 1980); total immersion (ASTM C67-00, 2000; ASTM C97/C97M-09, 2009); and evaporation curve (RILEM Test No. II-5, 1980). The material is wetted first by capillary absorption of water and second by total immersion for 24 hours; it is then dried until it no longer loses weight. The weight gain during absorption and immersion is an estimation of the amount of water the building materials can absorb when subjected to constant rain. The rate of weight loss during drying indicates the susceptibility of the material to damage from biocolonization, salt accumulation, and other factors.

These simple tests will allow conservators to assess the conditions of their buildings and monuments more readily without the need for expensive testing and equipment and thus aid in buildings and monuments' preservation.

## 1. EXAMINING CONDITION: SIMPLE MICROSCOPY

## JORGE OTERO AND A. ELENA CHAROLA

When examining a building to determine its surface condition, it is important to be able to view it at different magnifications, depending on the nature of the deterioration. For this purpose, microscopes, which include magnifying glasses, are necessary. What kind of magnification is required depends on the substrate being evaluated.

Any source of magnification can be useful to view small details of an object and may also serve to identify the main minerals in a stone, recognize deterioration patterns, and evaluate the condition of the observed material. The selection of the instrument depends on the type of materials to be viewed. For the case of building materials, the stereo and/or the digital microscope will probably be the most useful.

There are many kinds of microscopes, and they can be classified according to their type. The most basic type is the magnifying glass, by which the image is magnified by using a single lens. These glasses can range in magnification, from  $3 \times 10^{-2}$  to  $25 \times 10^{-2}$ , and they also may include a light source to illuminate the area in question and are not very expensive.

The most relevant type of microscope in the field of conservation is the stereoscopic microscope, which allows one to observe the object at magnifications between 10× and 200×. Recently, digital microscopes have been developed that offer a similar magnification range and have the advantage that they can be taken into the field (a building site or other structure being examined) and can record the photos straight into a computer or smartphone. Finally, a paper-based microscope, developed by Foldscope Instruments, is a promising magnifying tool owing to its performance and low cost.

Both optical and digital microscopes are useful tools that help conservation practitioners to characterize materials and their condition and to monitor the deterioration processes in historic substrates, either in the laboratory or on-site. The types of microscopes and some properties are summarized in Table 1.1.

**TABLE 1.1.** Properties and benefits of different types of magnification instruments. The check mark ( $\checkmark$ ) indicates a property or benefit that is typically available for a given instrument; PC: can be connected to computer; Micrograph: can take photos; \$ indicates relative costa; a dash (—) indicates the magnification or property is not available or applicable for that type of instrument.

Type of		<b>Usual magnification</b>			Other properties			Relative	
instrument	2×	10×	50×	100×	200×	Portable	РС	Micrograph	costa
Magnifying glass	✓	✓				✓			\$
Digital Microscope	$\checkmark$	$\checkmark$	$\checkmark$	$\checkmark$	$\checkmark$	$\checkmark$	$\checkmark$	✓	\$\$\$
Stereo Microscope	$\checkmark$	$\checkmark$	$\checkmark$	$\checkmark$	$\checkmark$			✓	\$\$\$
Foldscope	$\checkmark$	$\checkmark$	$\checkmark$	$\checkmark$		$\checkmark$	$\checkmark$	✓	\$

a\$ = inexpensive; \$\$\$ = expensive.

## **Equipment**

## **Magnifying Glass**

In conservation practice, a magnifying glass, also referred to as a hand lens, specifically provides help when examining a building or structure to identify the nature of the material (i.e., type of stone, brick, etc.), surface cohesion, deterioration patterns such as cracks, blistering, or crusts, as well as identification of biological growth or the presence of salts in the surface of the stone. A magnifying glass is a convex lens that is used to produce a magnified image of the detail of an object (Figure 1.1).



FIGURE 1.1. Magnifying glass with 10×, 20×, and 30× magnification (M20, Fancii Optics).

The highest magnifying power is usually obtained by putting the lens very close to one eye and moving both eye and lens together to obtain the best focus, usually a distance of 25 cm (10 inches) (Hecht, 1987: 186–188). A typical magnifying glass has a magnification between 2× and 10×, the human eye being 1×. This means that a 7 cm (2.7 in.) object at 2× total magnification power would appear to be 14 cm (5.5 in.). This is "low magnification" in comparison with other microscopes. Nonetheless, magnifying glasses can be very useful for observing surface details smaller than 0.63 cm (0.25 in.), which can be difficult for the human eye to detect unaided.

## Digital Microscope

The digital microscope is a variation of a traditional microscope; it uses optics and a digital camera to output an image to a monitor, sometimes by means of software running on a computer or a smartphone. A digital microscope imaging system typically includes four components:

- 1. Microscopy optical module
- 2. Data acquisition module
- 3. Digital image processing
- 4. Software control modules

The digital image is obtained by combining optical microscopy with digital processing technology that will be shown on a computer or a smartphone screen through the computer's software. Digital microscopes can range from very inexpensive USB microscopes, which are commercially available, to advanced industrial digital microscopes, some of which are wireless. Three factors account for the main difference between a low-cost USB digital microscope and the more sophisticated ones:

- 1. Image quality, which depends on the lens, the sensor, and the number of pixels collected
- 2. The number of magnification powers
- 3. The software for digital imaging processing

The last factor is usually the weakest aspect of the inexpensive digital microscopes; these usually come with simple software that can only record the image, whereas the advanced industrial digital microscopes include a good standard software that includes several functions useful for reporting, such as magnification power scale, comparison of images, and other features.

Digital microscopes are usually easy to use, and images can be stored and sequentially processed by digital processing technology. Magnification is typically claimed to be user adjustable from 10× to 200×, sometimes with a significant resolution. This degree of resolution is an advantage for conservation



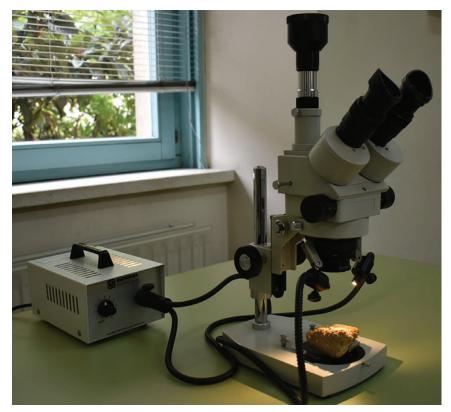
FIGURE 1.2. Digital microscope connected to a computer (Jiusion digital portable microscope).

applications because digital microscopes can provide significant help in several areas, such as mineral characterization and evaluation of deterioration patterns (Figure 1.2). Moreover, these USB microscopes are portable; thus they can be used in the field directly on the historic building.

### Stereomicroscope

The stereomicroscope is commonly used in conservation practice to observe small details in objects and any anomalies those objects may have; this use also applies to stone or other construction materials and their deterioration processes (Figure 1.3). The microscope uses visible light and has a system of lenses to generate various magnified images. The object is placed on the platen and viewed through the ocular lenses. The stereomicroscope has the advantage that the object is viewed in three dimensions. Stereomicroscopes usually provide a range of different objective lenses with different magnification powers, which usually range from 200× to 300×.

In conservation practice, the most commonly used magnification ranges between 10× and 200×, so that small details (between 0.3 mm and 3  $\mu$ m) of a stone surface can be observed; this range is useful for



**FIGURE 1.3.** An example of a stereomicroscope (Euromex zoom 4.5×-7× stereomicroscope).

the identification of the stone, mortar, and other building materials, as well as allowing the estimation of void sizes, aggregate cohesion, and the identification of deterioration patterns such as blistering, efflorescence, and biological growth. With some stereomicroscopes, a camera can be attached to capture the images observed through the eyepiece, commonly called "micrographs." The main drawback of optical microscopes is that they are not portable, so the sample has to be taken to the place that houses the microscope.

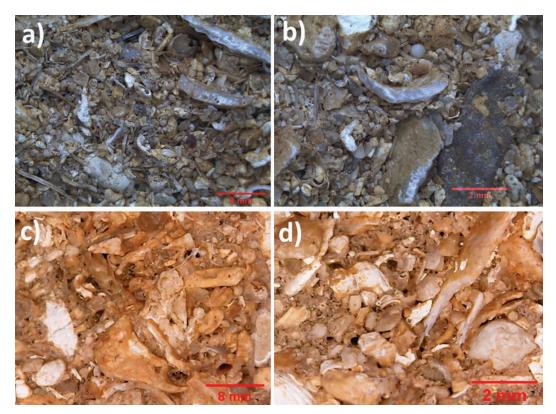
### Paper Microscope

An interesting variation of an optical microscope was recently developed at Stanford University (California, USA) by using paper as the support material for the optics. This Foldscope microscope or paper microscope, created by Foldscope Instruments, is an "ultra-affordable optical microscope" that is assembled from a punched sheet of cardstock, which includes a spherical glass lens, a light, a diffuser plane, and a watch battery that powers the LED light. The cost of production of each of those microscopes is less than US\$1, and it currently provides a good quality and low-cost tool for medical and biological science in communities around the world.

The image is obtained by observing transmitted light through the translucent material. The Foldscope microscope has a magnification from 2× to 140×, it is portable, and the images obtained can be saved and recorded by attaching it with a magnetic clip to a camera or a smartphone. In conservation, it can be useful to identify translucid organic materials, such as some types of biological materials, to study salt efflorescence, and to examine petrographic thin sections.

## **Example**

When observing a structure in place, it can be done with a magnifying lens and also, if possible, with a digital microscope. Figure 1.4 shows photos of the same stone that were taken at two magnifications (10× and 30×) with both a stereomicroscope and a digital microscope to allow comparison. A general examination performed using the stereomicroscope (Figure 1.4a,b) showed very good resolution for both magnifications; the stone is a biocalcarenite rock composed mainly of bioclasts and whole skeletal fossil remains of marine aquatic organisms such as marine bivalves, gastropods, rhodoliths,



**FIGURE 1.4.** Comparison of photos taken with a stereomicroscope (Euromex zoom 7×-4.5× stereomicroscope) and a digital camera (Nikon DS-Fi1), (a) 10× and (b) 30×; with those taken by an ultra-affordable digital microscope (Jiusion Digital portable microscope) bottom line, (c) approximately ×10 and (d) approximately ×30, of the same biocalcarenite stone.

echinoderms, and bryozoans. Carbonate lithoclasts were also observed together with a few quartz grains that are bound together by a fine-grained calcite (micrite). An examination with the ultra-affordable digital microscope showed significant lower resolution and image quality compared with the stereomicroscope. Furthermore, depending on the LED light quality of the digital microscope, it can cause some color variation, in this case a yellow-ish-orange shade, as observed in Figure 1.4c,d. Nonetheless, the ultra-affordable digital microscope can be useful to appreciate stone mineral morphology such whole skeletal fossils, quartz or calcites (Figure 1.4c), cracks, and voids, as well as grains attached to the matrix (Figure 1.4d). To be taken into consideration is that the digital microscope is portable, which may be useful for a first examination of a building.

## 2. INVESTIGATING SALT PROBLEMS: ION TEST STRIPS

## A. ELENA CHAROLA AND JORGE OTERO

Building materials frequently have salts present in them. Detection of the presence of salts is fundamental for identifying the cause of the deterioration pattern observed. These salts can originate from various sources (Charola, 2000; Bläuer and Rousset, 2014; Charola and Bläuer, 2015); some may be inherent to the stone itself, as is the case of those deposited in a marine environment or near a floodplain. Manufactured materials may also contain salts. For example, Portland cement may release significant quantities of sodium and potassium hydroxides, sulfates, and carbonates, whereas a dolomitic lime mortar may release magnesium hydroxide, carbonate, and hydrogen carbonate (Bläuer Böhm and Jäegers, 1997), and bricks may contain sodium sulfate if not appropriately fired (Charola and Rörig-Dalgaard, 2019)—all of which are water soluble.

Significant amounts of nonautochthonous salts can enter these porous inorganic materials once they are part of a building or structure as a result of water infiltrations, such as rising damp (i.e., water rising through the salt-containing soil in contact with the masonry wall), so that salts accumulate in the porous materials over time. Buildings may also have been used to keep salts in storage, such as common table salt, as for example the smokehouse in Colonial Williamsburg in the USA (Livingston and Taylor, 1998); or part of the structure may have been turned into stables for cattle or horses so that the nitrates and sulfates in their manure accumulated in the walls.

Salts can penetrate a structure from the ground, from air pollution, and from de-icing salts used in winter because they are capable of dissolving in water. Once the amount of water decreases, the salts will crystallize out and induce strains in the stone or brick matrix. Since the building is subjected to periodic wetting and drying, the repeated crystallization and dissolution will enhance the initial deterioration caused by the salts. Furthermore, depending on the atmospheric conditions, salts can migrate within the building (Charola, 2000).

It is important to identify the salts present in the material of the building in question. In general, the most common salts are sodium chloride (NaCl, halite), that is, common table salt, which is used as one of the de-icing salts; gypsum (CaSO<sub>4</sub>.2H<sub>2</sub>O), which results from the reaction of the sulfate from air pollutants with the calcium present in the stone or the mortar; and nitrates, such as niter (KNO<sub>3</sub>) or soda niter (NaNO<sub>3</sub>), which mostly result from biological activity. It is also common to find other salts, such as sylvite (KCI), sodium sulfate (hydrated or anhydrous), and epsomite. A more complete list of the most common mineral salts is given in Table 2.1.

TABLE 2.1. The most commonly found mineral salts in deteriorating masonry and concrete or cement mortars.

Mineral	Chemical formula	Name			
In deteriorating masonry					
Gypsum	CaSO <sub>4</sub> .2H <sub>2</sub> O	Calcium sulfate dihydrate			
Thenardite	Na <sub>2</sub> SO <sub>4</sub>	Sodium sulfate			
Mirabilite	Na <sub>2</sub> SO <sub>4</sub> .10H <sub>2</sub> O	Sodium sulfate decahydrate			
Epsomite	MgSO <sub>4</sub> .7H <sub>2</sub> O	Magnesium sulfate heptahydrate			
Halite	NaCl	Sodium chloride			
Sylvite	KCI	Potassium chloride			
Niter	KNO <sub>3</sub>	Potassium nitrate			
Soda niter	NaNO <sub>3</sub>	Sodium nitrate			
Brushite	CaHPO <sub>4</sub> .2H <sub>2</sub> O	Dicalcium phosphate dihydrate			
Hydromagnesite	3MgCO <sub>3</sub> .Mg(OH) <sub>2</sub> .3H <sub>2</sub> O	Magnesium carbonate, Mg hydroxide trihydrate			
Thermonatrite	Na <sub>2</sub> CO <sub>3•</sub> H <sub>2</sub> O	Sodium carbonate monohydrate			
Natron/Soda	Na <sub>2</sub> CO <sub>3</sub> .10H <sub>2</sub> O	Sodium carbonate decahydrate			
Calcite	CaCO <sub>3</sub>	Calcite			
	In deteriorating concrete o	or cement mortars			
Aphthitalite	$K_3Na(SO_4)_2$	Potassium sodium sulfate			
Trona	NaHCO <sub>3</sub> .Na <sub>2</sub> CO <sub>3</sub> .2H <sub>2</sub> O	Sodium carbonate bicarbonate dihydrate			
Ettringite	3CaO.Al <sub>2</sub> O <sub>3</sub> .3CaSO <sub>4</sub> .32H <sub>2</sub> O	Hydrous calcium aluminum sulfate			

## **Equipment**

Commercially available test strips for different ions operate similarly to the pH strips. They are very practical because apart from identifying the presence of the anion or cation in question, they can also provide a semiquantitative value of its concentration. These test strips are available for various ions, such as chloride (Cl<sup>-</sup>), sulfate (SO<sub>4</sub><sup>2-</sup>), nitrate (NO<sub>3</sub><sup>-</sup>), nitrite (NO<sub>2</sub><sup>-</sup>), phosphate (PO<sub>4</sub><sup>-3</sup>), and ammonium (NH<sub>4</sub><sup>+</sup>).

In general, cations are easier to identify than are anions. Please note that the most common cations are sodium (Na+), potassium (K+), calcium (Ca++), and magnesium (Mg++). A simple "tasting" of a grain from an efflorescence can determine whether the salt is sodium chloride (common table salt) or a magnesium salt because the latter tastes bitter. Magnesium sulfate can crystallize with different amounts of water, forming a monohydrate, a tetrahydrate, a pentahydrate, a hexahydrate, and finally a heptahydrate (epsomite). Therefore, the efflorescence can appear and disappear, depending on the relative humidity conditions.

For testing purposes, the presence of salt has to be evident on the building as an efflorescence. Please note that its presence will depend on the weather conditions: if it is very damp, the salts may be dissolved and not readily visible. Therefore, it is important to check the site on both dry and humid days.

To determine which anion is present, the anion strips should be used. Please note that the most useful strips, that is, for the most common anions, are those for chlorides (Cl<sup>-</sup>), sulfates ( $SO_4^{2^-}$ ), and nitrates ( $NO_3^-$ ). Phosphates can occur, but they are not as common, and the presence of ammonium ion ( $NH_4^+$ ) is not very common.

Given the cost of the ion strips, they are most useful to determine anions, which require more complicated laboratory tests to detect them. The concentration range (mg/L) for which an ion can be measured depends on the ion and the particular brand of the strip. One of the drawbacks of these test strips is their high cost, so their use for ion identification purposes should be limited for in situ testing. If necessary, they can be used in the laboratory for a semiquantitative determination in a given sample.

## **Field Testing**

To test for the presence of salt, one option is to wet the paper strip and apply it to the efflorescence (Figure 2.1a-c). Another option is to detach some efflorescence onto a plastic glass slide or into a petri dish, mix it with a drop of water, insert the paper strip in the solution, and leave it for several minutes until the strip has finished changing color. Upon completion of the color change in the strip, compare its color to that of the color and concentration scale provided on the tube or box of the strips (Figure 2.1d). Since the amount of water is not measured, the obtained concentration is just approximate.

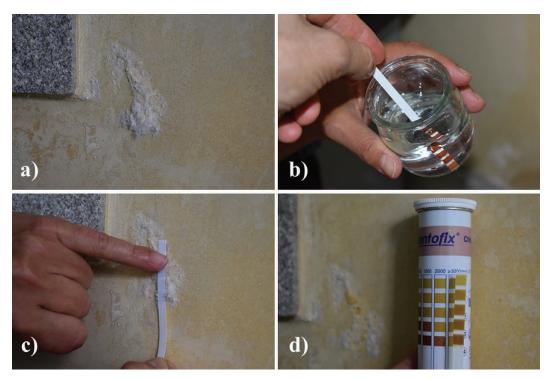


FIGURE 2.1. Use of ion test strip. (a) Salt efflorescence on a wall; (b) salt strip is prewet before the test; (c) a chloride ion testing strip is applied directly to the salt efflorescence on the wall; (d) the test strip is compared with the strip on the tube to approximate concentration. The concentration scale on the tube ranges between 0, 500, 1,000, 1,500, 2,000, and ≥3,000 mg/L of chloride ion, and one has to match the color of the strip used to one of those on the tube. In this case, the concentration was approximately 2,000 mg/L, which means that there are >2g/L of chloride ions in solution, which is a significant amount.

## **Laboratory Semiguantitative Concentration** Determination

To obtain a semiquantitative evaluation of the salt concentration, the efflorescence needs to be carefully scraped off the surface of the building or monument. Enough sample must be available for weighing; this means that at least 0.5 g of the sample is necessary for a nearly pure efflorescence or 1 g if it also contains powder of the deteriorating material. The steps to follow are listed below:

- 1. The weighed sample—remember to subtract the weight of the container—is put into a small beaker and dissolved in water. (If you have a powdered sample taken from the surface of a deteriorating stone/render/brick, only the salt will go into solution, and there will be a residue. In this case, the powdered sample should be left in water for at least an hour with occasional stirring).
- 2. This solution is diluted to obtain a given volume (e.g., 10 mL, 50 mL, etc.), either in a graduated cylinder or in a volumetric flask depending on the precision required. Record this volume, which contains all the ions of your sample.

- 3. An aliquot of this solution is placed in a small beaker, and the test strip immersed or drops of the solution are put onto the strip. (Read the instructions for each type of test strip.)
- 4. Once the color has developed, the concentration of the ion in question is given by the test strip. Please note that some test strips give the concentration of the ion itself (i.e., NO<sub>3</sub>-), whereas others give it as a compound (i.e., NaCl). Also note that some may give the concentration in mg/L (ppm), some in g/L.

The concentration of the ion in the sample is then calculated as follows ( $V_{SOlution}$  is volume of solution):

$$lon (g/g) \% = \frac{strip \ reading \ (mg/L) \times V_{solution} \ (L) \times 100}{weight_{sample} \ (g) \times 1,000 \ mg/g} \ .$$

If the concentration of the ion in question is too high, a dilution must be prepared from the solution, and this dilution must be accounted for in the calculation. Note that in this case the volume of the aliquot ( $V_{aliquot}$ ), as well as the volume of the dilute solution  $(V_{dilution})$ , must be measured exactly. An unmeasured aliquot of this dilution is taken to make the actual measurement.

$$lon\left(\text{g/g}\right) = \frac{\textit{strip reading}\left(\text{mg/L}\right) \times V_{\textit{solution}}\left(\text{L}\right) \times V_{\textit{dilution}}\left(\text{mL}\right)}{V_{\textit{aliquot}}\left(\text{mL}\right) \times \textit{weight}_{\textit{sample}}\left(\text{g}\right) \times 1,000 \text{ mg/g}}.$$

Note: Technically, an aliquot means a part of a number or quantity that will divide it without a remainder; thus, 5 is an aliquot part of 15. In general, it means a measured smaller volume of a larger volume. For use with the strip, the aliquot does not have to be measured; however, if a dilution has to be prepared, then the volume of the aliquot must be known exactly as well as the volume of the original solution and of the dilution.

## **Final Remarks**

The salt test strips serve to identify the presence of soluble salts on the surface of a structure by confirming the presence of anions and/or cations. Strips will not identify the actual salt, which needs a laboratory procedure for its identification, such as microscopy or X-ray diffraction, but they can provide an estimate of the concentration of the ions present.

## 3. ASSESSING SURFACE COHESION: THE SCOTCH TAPE TEST

## JORGE OTERO AND A. ELENA CHAROLA

The "Scotch Tape" test is useful for determining the surface deterioration of a stone/brick sample as well as for evaluating the effectiveness of a consolidation treatment. This test, also known as the "peeling test," was introduced into the field of conservation by G. Torraca and P. Mora in the 1960s (Torraca and Mora, 1965) and has been widely used for over 60 years in conservation practice. The method measures the amount of detached material that adheres to the tape and is usually carried out before and after any conservation treatment or on weathered and sound stone/brick. The test follows the ASTM D3359-O2 standard and the recommendations established by Drdácký et al. (2012). The main objective of this test is to evaluate the surface cohesion of stone or brick. This is important because surface cohesion provides an idea of the surface deterioration of the material in question.

In this simple test, a strip of double-sided pressure-sensitive adhesive, such as Scotch brand tape, previously weighed, is applied to the surface to be evaluated, ensuring that the tape is totally adherent and then pulled off. Loose powder and grains of the surface will remain attached to the tape. The tape is then weighed again, and the result expressed as mg/cm². Several strips of the tape need to be applied to different areas of the surface to obtain a useful number of data points. It is important to carry out the test in several different areas because if the same area is tested again, the released material will obviously decrease. The results of the detached material can be analyzed either by weighing the released material following the ASTM D3359-02 standard or by visual examination with a digital microscope or a stereomicroscope (chapter 1), following the ASTM 4214-97 standard to evaluate the number and type of particles detached. The visual evaluation will provide a good enough approximation regarding the deterioration condition or the effectiveness of a conservation treatment.

## **Equipment**

- · Double-sided tape, preferably 2 cm width
- Balance with a ±0.001 g sensitivity
- Graph paper
- Appropriate boxes or plastic sampling bags with a closing pressure zip to carry the tape with the attached powder and grains to a laboratory for weighing
- Note: If a balance is not available, the tapes can be examined with a digital microscope or stereomicroscope, and an approximate visual evaluation can be carried out.

## **Test Procedures**

## **Tape Preparation**

The tape strips are prepared for the test as follows:

- 1. From the roll of double-sided tape, cut nine strips measuring at least 8 cm $^2$  (a strip of 4 × 2 cm) and having the exact same dimensions.
- 2. Press one side of each tape strip to a graph paper. Leave the protective sheet of the other adhesive side in place. Then both the tape and graph paper are cut to include an extra 1 cm of the graph paper at one end of the strip, which will be used to handle the sample strip (see Figure 3.1a). Since the graph paper is scaled, all free spaces must also present the exact same dimensions.
- 3. The nine strips are all put together in a plastic bag or a box with a hermetical cover and weighed to the milligram. The weight is divided by nine and then by the strip surface (e.g., 8 cm²) since all strips should be exactly equal in size and therefore in weight. Please note that other procedures and tape sizes have also been recommended (Drdácký et al., 2012, 2015; Drdácký and Slízková, 2015).

## **Peeling Test**

The Scotch Tape Test is carried out in situ as follows:

- Select the area of the building where the test is to be carried out. This area must be dry and reasonably clean before the test. Since the measurements might be influenced by the relative humidity, similar environmental conditions must be used for comparing the results of different tape tests.
- 2. The tape strip is affixed to the surface of the material (Figure 3.1b,c). Once it is attached to the surface, it is recommended to apply an even pressure with a finger and repeat this up to six times to ensure the tape's complete adhesion to the surface. It is very important to apply similar pressure and an equal number of applications of pressure to all strips to have similar conditions.

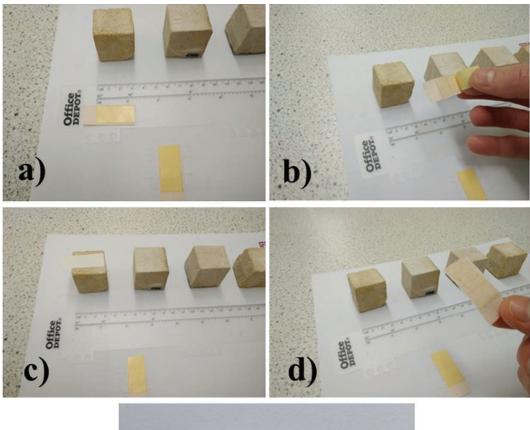




FIGURE 3.1. Scotch Tape test procedure. (a) One side of the double-sided tape is glued to the graph paper and a 1 cm<sup>2</sup> area of the latter is free of tape (left of the strip); (b) the protective sheet of the adhesive layer is released; (c) the tape is applied to the material; (d) the tape is pulled off, and the attached grains from the material surface are visible; (e) a detail of the detached grains attached to the tape is shown.

- 3. After approximately 1 minute, the strip is removed by pulling it off steadily and carefully by the uncoated end tab so that the strip does not lose any of the attached grains. It is commonly accepted that the strip must be withdrawn at a rate of about 10 mm/s and at an angle of 90° (Drdácký et al., 2015).
- 4. Once the strip is completely released, the strip and its protective sheet should be put into the plastic bag with a good seal or the hermetically closed box

- (Figure 3.1d). This procedure is repeated for all of the nine strips, which should be kept in the same plastic bag or box.
- 5. The bag (or box) with all the tape strips (which also include the aggregates attached to them) and protective sheets are weighed together on the balance, and their weight is compared with that of the same strips prior to the test; the difference in weight between the initial weighing and the final weighing corresponds to the released surface material.

## **Results and Data Presentation**

The data should be noted as reported in Table 3.1.

**TABLE 3.1.** Results of tape test applied to a weathered sandstone surface and then repeated after a consolidation. Tape strips measure  $4 \times 2$  cm  $(8 \text{ cm}^2)$ ;  $\Delta W$  (%) = (initial value – post-treatment value)  $\times$  100 / (initial value); n/a indicates measure not applicable.

	Released material	
Surface condition	(mg/cm²)	ΔW (%)
Untreated sandstone	84.59	n/a
Treated sandstone	21.90	74.11

The percent change in weight ( $\Delta W$ ) is calculated as follows:

$$\Delta W$$
 (%) =  $\frac{(initial\ value\ -\ post-treatment\ value) \times 100}{initial\ value}$ .

When several treatments are applied, the data can be reported as in Table 3.2.

**TABLE 3.2.** Chart with results of our tape test. Tape strips were applied to an untreated sandstone surface and again after consolidation treatments with three different products: tetraethyl orthosilicate (TEOS), an acrylic resin, and hydroxyapatite. Nine tape strips were applied, each measuring  $4 \times 2$  cm  $(8 \text{ cm}^2)$ , giving a total of  $72 \text{ cm}^2$  for all strips.

	•	plastic bag/ ne samples	_ Weight	Released
Sample treatment	Before peeling (g)	After peeling (g)	difference (mg)	material (mg/cm²)
Untreated sandstone	0.421	0.914	493	6.84
Treated with TEOS	0.416	0.557	141	1.96
Treated with an acrylic resin	0.428	0.685	257	3.57
Treated with hydroxyapatite	0.425	0.483	58	0.80

Date and time:a

Climatic conditions: temperature, relative humidity, wind, etc.<sup>a</sup>

<sup>&</sup>lt;sup>a</sup>Additional data that should be recorded for optimal evaluation of the tests.

And to compare the before and after treatment conditions, the data can be presented as in Table 3.3.

**TABLE 3.3.** Results of tape test applied to a weathered sandstone surface and then repeated after three consolidation treatments. Abbreviations:  $\Delta W$  = (initial untreated value - posttreatment value)/initial value; n/a = not applicable; TEOS = tetraethyl orthosilicate.

Released material		
Sample treatment	(mg/cm²)	ΔW (%)
Untreated sandstone	6.84	n/a
Treated with TEOS	1.96	71.72
Treated with an acrylic resin	3.57	47.81
Treated with hydroxyapatite	0.80	88.30

When no balance is available with the required sensitivity (to the milligram), the strips of tape can be examined by naked eye or under a digital or stereo microscope (chapter 1). The evaluation is only approximate but serves as a first estimation of the surface condition of the stone and is particularly useful to test areas that can be treated with different consolidants for comparison.

In this example, the sandstone samples treated by hydroxyapatite yielded the highest reduction of released material (88.3 % reduction), followed by samples treated by TEOS (tetraethyl orthosilicate; 71.79%) and acrylic resin (47.81%). These data serve to evaluate potential consolidation treatments and their effectiveness in providing surface cohesion to disintegrated areas. The tape test can be also useful to compare the level of weathering in a structure between more and less deteriorated areas.

# 4. INVESTIGATING DIFFERENTIAL WEATHERING: THE RILEM TUBE WATER ABSORPTION TEST

## A. ELENA CHAROLA AND JORGE OTERO

The main objective of this test is to study the capillary water absorption velocity of a surface, which will help in evaluating the moisture content in the material. This test is usually carried out when first appraising a structure to determine degrees of weathering. Eventually, if a conservation treatment is applied, it should be repeated to determine whether the treatment significantly changed the material's water absorption. Since the RILEM tube is portable, the test can be carried out either in the laboratory on treated samples or on-site before and after treatment. (RILEM is the acronym for Réunion Internationale des Laboratoires D'essais et de Recherches sur les Matériaux et les Constructions.)

Tests of capillary water absorption of stone surfaces on buildings or monuments can be carried out in situ using the RILEM or Karsten tube (Wendler and Snethlage, 1989). Field testing allows a comparison of the different conditions of stone in a building since weathering is not usually homogeneous. In general, this test is carried out in several areas of the building's surface to test different levels of weathering, which can be compared with a sound surface area (i.e., an unweathered surface) on the building. In general, the surfaces with higher capillary water absorption rates correspond to those with higher degrees of weathering. The approach can also be used to test potential protective measures, such as application of a consolidant or water repellent, as well as evaluating a cleaning intervention.

## **Equipment**

The RILEM Test Method No. II.4 (1980:200-204) uses a glass or plastic tube that is applied to the surface of the stone to be measured and then filled with

water; originally the tube held 4 mL of water, and currently it holds 5 mL. This tube can be obtained online as part of a kit (e.g., RILEM Water Penetration Test Kit) or from other sites at more affordable prices. The amount of water absorbed under low pressure for a given time allows comparison of the condition of the various stones tested. There are two variants of the tube: one for vertical surfaces and one for horizontal ones. The tube is attached to the surface using a putty, and then it is filled with de-ionized water. The time it takes for the water to be absorbed by the substrate is measured, as subsequently described.

For the horizontal tube, the cylindrical lower part that is attached to the stone has a diameter of 2.5 cm with a height of 2.5 cm, and the graduated section of the tube has a diameter of 0.84 cm and a height of 10.8 cm. In the case of the vertical tube, the same dimensions apply (for a diagram of these tubes see RILEM Test Method No. II.4., 2015). The top of the tube has a conical opening to facilitate filling it with water. The tube is graduated in milliliters (mL) from 0 mL at the top to 5 mL close to the base.

## **Test Procedure**

The following items are required for carrying out the test:

- RILEM tube
- Water repellent putty
- · Chronometer, a watch with second hand, or a smartphone with a stopwatch
- De-ionized water dispenser

Ensure that the surface to which the tube is to be attached is dry, smooth, and not disaggregating. To attach the tube, put the putty around the flat edges of the tube. Take care that no putty is introduced into the interior area where the water will be. Once the tube is attached to the wall (Figure 4.1), fill it up with de-ionized water to the 0 graduation at the top and record the time. Make sure that there is no leak around the attachment area. Take note of the time when the material has absorbed 1 mL, then 2, 3, 4, and 5 mL, at which point the test is completed. At least three tests should be run in the same area of the building so as to obtain an average value of the absorption velocity. Once the test finishes, the tube and putty are smoothly pulled from the wall; it is important to remove as much as possible excess putty from the wall.

In some instances, the stone may take quite some time to absorb the water, in which case it makes sense to use smaller gradations (the scale on the tube has gradations for each milliliter) to measure the water absorption. The absorption rate is dependent on the nature and condition of the stone to be measured.

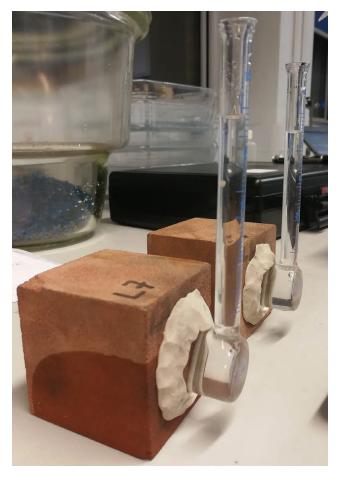


FIGURE 4.1. RILEM tubes installed on cubic samples in the laboratory for eventual comparison.

## **Results and Data Presentation**

Data should be recorded as shown in Table 4.1:

**TABLE 4.1.** Water absorption data collected and the calculated absorption rate.

Volume absorbed (mL)	Measured time	Time (min)	Absorption rate (mL/min)
1	1 min, 55 sec	1.92	0.52
2	3 min, 34 sec	3.57	0.56
3	5 min, 40 sec	5.67	0.53
4	7 min, 10 sec	7.16	0.56
Average	[Not calculated]	[Not calculated]	0.54

Date and time:a

Climatic conditions: temperature, relative humidity, wind, etc.a

<sup>&</sup>lt;sup>a</sup>Additional data that should be recorded for optimal evaluation of the tests.

Figure 4.2 shows the data plotted as water absorption versus time.

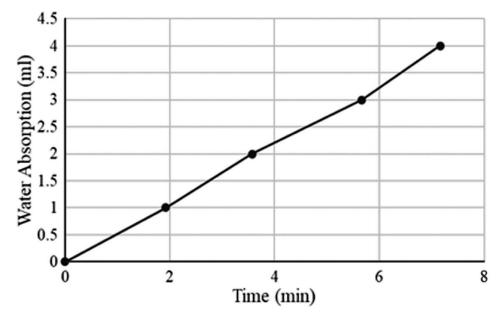


FIGURE 4.2. Plot of the data obtained. The slope of the line provides the water absorption rate.

The slope can also be estimated using the following formula:

Slope = water absorption rate = 
$$\frac{\text{final volume (mL)}}{\text{final time (min)}}$$
.

For the example shown in Figure 4.2, the final volume (4 mL) was divided by final time (7.16 min), giving an approximate slope of 0.56 mL/min, which is an approximation of the value calculated by averaging all the data in Table 4.1 (0.54 mL/min).

If the material is not very absorbent, for example, a dense stone or the surface has a crust, the initial absorption may be very slow, which may be misleading as to its actual absorption capacity. One way of measuring absorption in this case is to start taking a reading every 5 minutes until close to the 5 mL limit. The data can be recorded as shown in Table 4.2. It is clear that water absorption does not have a regular rate, and it can be estimated that the surface has the lowest absorption.

**TABLE 4.2.** Water absorption data collected and calculated absorption rate for the case of a dense stone material.

Measured time (min)	Volume absorbed (mL)	Absorption velocity (mL/min)
5	1.2	0.24
10	3.7	0.37
15	4.35	0.29
Average	[Not calculated]	0.30

Date and time:a

Climatic conditions: temperature, relative humidity, wind, etc.a

<sup>&</sup>lt;sup>a</sup>Additional data that should be recorded for optimal evaluation of the tests.

## 5. COMPARING MATERIAL COMPATIBILITY: THE WATER VAPOR TRANSMISSION TEST

## JORGE OTERO AND A. ELENA CHAROLA

The main objective of this test is to compare materials such as stone, bricks, and mortars, as well as protective products such as water repellents and paints to be able to select the protective product having a similar permeability or water vapor transmission rate (WVTR; Hern and Snethlage, 1992; DeFreece and Charola, 2007; Galván et al., 2014; Liu and Charola, 2014). This is a critical point because the ideal situation would be that a paint, protective treatment, or render should have a WVTR as similar as possible to that of the material to which it is to be applied.

The WVTR of a material is critical for determining the resistance to water evaporation when the material is wet, either by capillary absorption from the ground or from the application of a protective treatment that may reduce the evaporation of liquid water. When the material retains moisture, problems such as dampness in the interior of the building may appear, and biocolonization may occur on the exterior. For this reason, a test has been devised to allow comparing the permeability of the material itself and the material with the application of a paint or a water repellent.

There are two approaches that can be followed for this test, the "wet-cup," later referred to as "water method," and the "dry-cup," or "desiccant method" (ASTM E 96/E 96M-05). The test described below has been simplified to determine the WVTR by means of the "wet-cup" procedure, by which a disk of the material in question—either brick, stone, or other material—is sealed over a beaker filled with a given amount of water. The beaker is placed in a closed, dry environment and weighed regularly to monitor its change in weight, as water vapor will permeate through the stone and be absorbed by the drying material used to keep the environment dry.

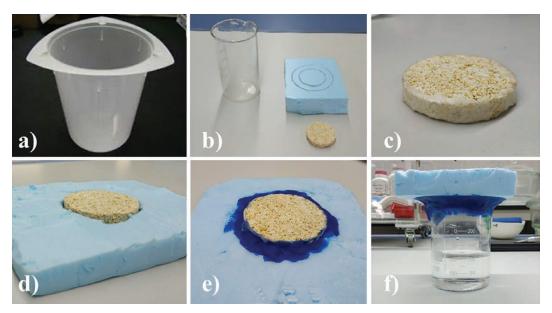
## **Equipment**

- 300 mL plastic or glass beakers; alternatively, 250 mL tricornered plastic beakers with a diameter of 7.5 cm, having a 0.2 cm wide interior ledge approximately 0.5 cm down from the top where the sample can be wedged in (Jacob and Weiss, 1989)
- Balance with a ±0.01 g sensitivity
- · De-ionized water
- Any impermeable and malleable material: in the experiment described, a
  Polyurethane sheet (4 cm thick) for holding the disk sample, parafilm for sealing
  the sides of the disk, and putty for sealing the disk to the sheeting and the
  beaker
- An air-tight container, large enough to accommodate all samples to be tested
  and with a fairly tight cover to keep the samples at close to 10% relative humidity
  (RH) using oven-dried silica gel (dried approximately for 24 hours at about 30°C
  until constant weight); should silica gel not be available, the closed environment
  can be kept at low RH (about 10%) with oven-dried rice (dried about 24 hours
  at about 30°C until constant weight)
- Hygrometer to monitor changes in the container; alternatively, a humidity card indicator, such as the SCS 4HIC100 from Digi-key Electronics (https://www. digikey.com/products/en?keywords=SCS%204HIC100%20, accessed 18 June 2020); please note that the temperature should also be constant during the test
- Cut disks of the sample(s) sized depending on the beaker used for the test: when using the 300 mL beaker, a 1 cm thick disk with a 5.3 cm diameter was prepared; if the 250 mL tricornered beaker is used (Jacob and Weiss, 1989), a 1.9 cm thick disk and 6.9 cm diameter is required (see Figure 5.1a)

Note: Alternatively, the discs can be cut to the size of the beaker mouth and the edges of the disks lined with parafilm; then wax can be used to seal them to the beaker mouth (Jacob and Weiss, 1989). When comparing different materials, or materials treated with a conservation product, all disks should present the same thickness and diameter to allow comparison. It is suggested that the test be carried out on three samples per each material to be tested to allow comparison of the results for each type of sample.

## **Test Procedure**

- 1. Before running the test, the silica gel or rice is oven dried for 24 hours. Once dry, either material is introduced into a beaker and placed inside a hermetic container to stabilize the relative humidity to approximately 10%.
- 2. Measure thickness and diameter of the disk sample, label it, and then weigh it when it is completely dry.
- 3. In our experiment, the holder for the sample disk was made by cutting a round hole from a polyurethane sheet with a thickness similar to the disk (1 cm), and



**FIGURE 5.1.** Equipment for water vapor transmission test. (a) A tricornered plastic beaker that has a ledge on which the sample can rest (Jacob and Weiss, 1989). (b) Calcarenite stone sample disk, piece of polyurethane to be cut where the disk is to be inserted, and the glass beaker that will contain water. (c) Sample disk with the full thickness of its edge protected by parafilm. (d) Disk inserted into the polyurethane sheet. (e) Disk secured in polyurethane with putty. (f) View of the "wet cup"—the beaker with water and the sealed polyurethane sheet in which the sample is secured.

the hole in the sheet had the same diameter as our sample (Figure 5.1b). The edge of the disk sample must be sealed with parafilm or a plastic material around its entire circumference (Figure 5.1c).

- 4. The disk sample is then placed into the space in the polyurethane sheet (Figure 5.1d), and the space between the sample disk and polyurethane is sealed with putty (Figure 5.1e).
- 5. Fill the beaker with 100 mL of water, and then attach the disk in the polyurethane sheet to the beaker with additional putty (Figure 5.1f). Make sure the putty completely seals the space between the beaker and the polyurethane. Once this beaker is ready, it is labeled, weighed, and placed inside the covered hermetic container, and the time is noted.
- 6. Weigh the beaker regularly, for example, every 12 hours. Usually, the weight is monitored about once or twice per day, depending on the sample. The weight of the cup will decrease as the experiment progresses, indicating the amount of water that passed through the material (e.g., stone, brick, mortar) from the wet environment (inside the beaker) to the dry environment in the container (about 10% RH). The test finishes when a constant slope of the reduction of weight (g/cm²) per hour is obtained (see Figure 5.2), which means that the WVTR has been determined.
- 7. The following data should be recorded: time (hours), weight (g), weight lost (g), and weight loss by area (g/cm²).

The decrease in weight is obviously dependent on the nature and condition of the material to be measured. Very permeable bricks or stone will decrease in weight more quickly than material that is less permeable to water vapor. In general, a minimum of five measurements should be taken in order to obtain the reliable WVTR.

#### **Results and Data Presentation**

Data were collected as shown in Table 5.1, and a graph was drawn as shown in Figure 5.2.

**TABLE 5.1.** Data collected on water loss (by weight) over time for the calcarenite-disk assembly illustrated in Figure 5.1. Weight measurements are for the sealed disk-beaker assembly. (Only one experimental sample is reported here as an example.) Original weight of disk = 35.57 g; disk diameter = 5.3 cm (0.053 m).

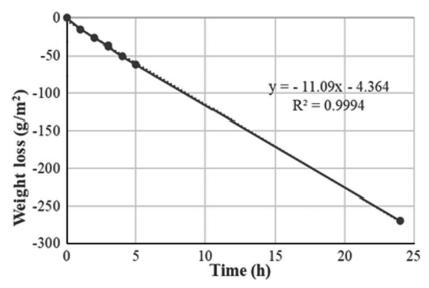
Elapsed time (h)	Measured weight (g)	Cumulative water weight lost (g)	Cumulative weight loss by area (g/m²) <sup>a</sup>
0	285.377	0	0
1	285.342	-0.035	-15.7
2	285.316	-0.061	-27.4
3	285.293	-0.084	-37.7
4	285.264	-O.113	-50.7
5	285.239	-0.138	-61.9
24	284.776	-0.601	-269.7

<sup>&</sup>lt;sup>a</sup>The surface area (A, in m<sup>2</sup>) was calculated with the standard formula,  $A = \pi r^2$ .

The WVTR is given by the slope of the obtained line, which in this case was -11.09 g/m<sup>2</sup> per hour;  $R^2 = 0.9994$ , showing that the data were consistent. The  $R^2$  value corresponds to the proportion of the variance of the dependent variable that is predictable from the independent variable; the closer the value is to 1, the stronger the correlation between the dependent and independent variables.

The slope and  $R^2$  values can also be calculated directly from the data in Table 5.1 using Microsoft Excel software; the data used for the y-axis are the weight loss calculations, and the x-axis is time. With Excel, the  $R^2$  function is calculated separately.

The point of this test is to compare different protective products, such as paints, water repellents, or others, that are being considered to prevent deterioration as well as to identify the product that will affect the WVTR minimally, since it is important to maintain a similar behavior for the treated material.



**FIGURE 5.2.** Water vapor transmission rate (WVTR) graph for the calcarenite stone. The equation of the trendline was provided by the software (Trendline, Microsoft Excel) and provided the slope (–11.09) of the line.

As an illustration, three different stone samples were used: the mentioned calcarenite stone, which is a limestone that contains over 50% detrital sand-sized particles (0.0625 to 2 mm in diameter) of carbonate grains; a sandstone, which is primarily constituted by sand grains; and a granite. To complement these, the WVTR of the polyurethane sheet, that is, a piece of the same polyurethane sheet used in the test, was evaluated for its impermeability. Table 5.2 gives the comparison between WVTR values of the different materials, and Figure 5.3 shows the graphic results for these tests.

**TABLE 5.2.** Water vapor transmission rate (WVTR) obtained from the slope of the regressions plotted in Figure 5.3;  $R^2$  is a statistical measure of how close the data are to the fitted regression line.

Sample	WVTR (g/m²/h)	R <sup>2</sup>
Calcarenite	11.09	0.9994
Sandstone	10.67	0.9993
Granite	1.54	09636
Polyurethane	0.25	0.9697

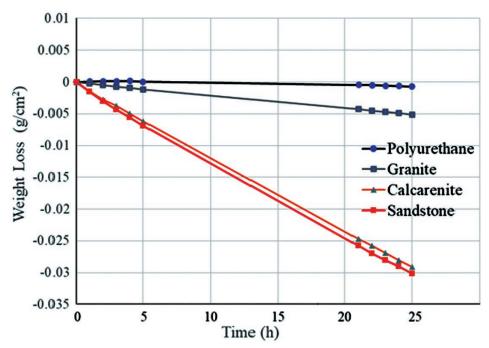


FIGURE 5.3. Comparison of the WVTR for three different stones, a calcarenite, a sandstone, and a granite; for each three samples were tested, and the average calculated and shown on the graph. The polyurethane material that was used to support the stone disks was also tested to confirm that it is practically impervious.

# 6. THREE SEQUENTIAL TESTS FOR MATERIALS EVALUATION: CAPILLARY WATER ABSORPTION COEFFICIENT, TOTAL IMMERSION, AND EVAPORATION CURVES

#### A. ELENA CHAROLA AND JORGE OTERO

One of the requisites in architectural preservation is material compatibility. The problem of mismatched materials has led to much damage as evidenced by the inappropriate use of Portland cement mortars or renders on historic brick structures as well as patching materials that are used to complete stone masonry or even concrete (Weinstein and Capen, 2014). One of the key points that give rise to these problems is the difference in porosity of the two materials (Binda and Baronio, 1985); hence, it is critical to assess the compatibility of materials as a function of their porosity. Although there are several methods to evaluate porosity, a practical approach is needed. The procedure described here serves to evaluate water absorption and loss characteristics of materials and to assess compatibility of different materials.

The procedure described below has been developed based on following sequentially simple tests: capillary water absorption (UNI 10859, 2000), apparent porosity (ASTM C67-00, 2000; ASTM C97/C97M-09, 2009), and drying or evaporation curves (RILEM Test Method No. II.5. 1980; DIN EN 16322, 2013). Some of these tests have been modified and simplified so that they can be carried out with a minimum of equipment and on various specimens.

The porosity of the material defines how water, in either liquid or vapor form, will circulate through the pores (Charola and Wendler, 2015). Table 6.1 identifies the various mechanisms of moisture transfer within porous materials of different pore diameters.

**TABLE 6.1.** Mechanisms of moisture transfer depending upon the pore size (adapted from Charola and Wendler, 2015): Ø is pore diameter.

Macropores		Micropores	Nanoj	oores		
Ø > 1 mm	1 mm-10 µm	10 μm-1 μm	1 μm-0.1 μm	100 nm-10 nm	< 10 nm	
Liquid water	Capi	llary	Wa	ater vapor adsorption		
flow	absorption			nd surface diffusi	on	
	Water vapo	or diffusion		Capillary co	ndensation	

Liquid water will enter a structure mainly by (a) capillary rise of moisture from the ground or (b) by gravitational infiltration of rainwater from above. The rise of groundwater results from capillary absorption via micropores having diameters between 1 mm and 1  $\mu$ m. Capillary rise occurs faster in smaller pores, but less water can enter them.

The evaporation of water from larger capillary pores is faster than for the smaller ones. For the smallest pores (those between 1  $\mu$ m to <10 nm), the mechanism of water movement is based on adsorption of water vapor on the pore wall, forming an ordered structure. This water layer will attract more moisture, and surface diffusion will occur. As more water is adsorbed, the water layers lose their structure, reaching the disordered condition of liquid water.

When the material (stone, brick, mortar, etc.) dries, three stages can be differentiated: the initial water evaporation that occurs at the surface of the sample; then a mixed evaporation from both surface and the pores closest to the surface; and, finally the evaporation from the interior of the stone.

When evaluating the performance of a material, three tests can be used in sequence to determine how fast the material absorbs water by capillarity, how much water can be absorbed by total immersion in water, and how fast the water evaporates. This information is crucial to understanding the behavior of the material in the presence of water as well as serving to determine its porosity.

# Methodology

These three tests, which in general are carried out separately, were sequenced to follow each other to simplify testing.

The tests should be carried out on regular-shaped samples, such as  $5 \times 5 \times 5$  cm cubes, that should be oven dried at  $60^{\circ}$ C ( $140^{\circ}$ F) to constant weight (usually about 24 hours). The best place to have these cubes cut is an outfit that deals with kitchen counters and/or stone floor tiles.

In principle, at least three samples of each type should be used: three control samples, three treated samples, and three of any other samples. The weight of the dry samples should be recorded. A balance with a sensitivity of  $\pm 0.01$  g is sufficient for these samples.





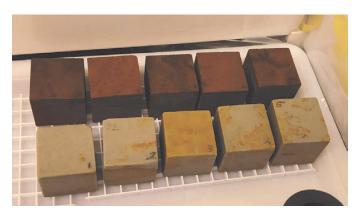


FIGURE 6.1. The three tests: (top) capillary water absorption, (center) total immersion, and (bottom) drying. Please note that, except for the drying stage, the containers for capillary water absorption and total immersion are normally covered to avoid water evaporation.

The sequence of the three tests is as follows (Figure 6.1):

- 1. Capillary water absorption
- 2. Total water immersion
- 3. Drying or evaporation curves

# **Equipment**

- Oven to dry the sample (a kitchen oven can be used if necessary, but it needs to be calibrated with a thermometer)
- Balance with ±0.01 g sensitivity
- · Chronometer or watch with second hand
- Plastic container large enough to accommodate all samples to be tested and with a tight cover
- Substrate such as glass beads, glass rods, filter paper, or cotton sponge cloth to line the bottom of the container
- · De-ionized water
- Beakers large enough to fit one sample
- · Racks to allow drying the samples

#### **Test Procedures**

#### **Capillary Water Absorption Coefficient**

After noting the weight of the dry sample ( $W_{\rm d}$ ), rest the sample on the chosen substrate material in a container-either glass beads, a pad of filter paper, or a cotton sponge cloth. Add de-ionized water to the container until the water level reaches the bottom of the sample, taking care that the water does not touch the sides of the sample. It is critical to record the time. Cover the container as hermetically as possible. As the samples absorb water, check periodically that the water level in the container remains fairly constant and, when necessary, add water to keep it so.

The sample needs to be weighed periodically as it absorbs water. The time between measurements needs to be adjusted to the porosity of the sample. As a rule of thumb, readings must be taken closely together at the beginning and then spaced out as absorption decreases. It is advisable to do a first short run weighing the sample every 5 minutes for the first half hour, then every 15 minutes for the second half hour to gauge the appropriate timing for the complete run.

To weigh the sample, take it out of the container, pat the bottom of the sample dry with a paper towel, and put it on the balance. Take the reading as fast as possible and return the sample to the container, making sure to close it again. Repeat at appropriate intervals.

It is important to record the data as they are acquired. For this purpose, prepare a blank table with six columns to record (1) actual time of measurement; (2) cumulative time (minutes); (3) square root of time (seconds<sup>0.5</sup>); (4) weight of sample at time t,  $W_t$  (g); (5) amount of water absorbed at time t,  $U_t$  (g):

 $U_{\rm t} = W_{\rm t} - W_{\rm d}$ , where  $W_{\rm d}$  = weight of dry sample; and (6) the amount of water absorbed per unit area,  $M_{\rm i}$  (g/cm<sup>2</sup>):  $M_{\rm i} = U_{\rm t}/S$ , where S is the absorbing surface area. For a 5 cm cube, S would be 25 cm<sup>2</sup> (see Table 6.2).

**TABLE 6.2.** Data for capillary water absorption test for sample 1 (an example). Amount of water absorbed per unit area  $(M_i) = U_t/S$ , where  $(U_t) =$  amount of water absorbed (change in weight) and S = absorbing surface area (25 cm²); a dash (—) = no measurement or calculation made.

Actual time	Cumulative time (min)	Square root of time (sec <sup>0.5</sup> )	Weight at time ( $W_t$ ) (g)	Absorbed water (U <sub>t</sub> ) (g)	Water absorbed per unit area ( <i>M</i> <sub>i</sub> ) (g/cm²)
08:00	0	Ο	284.37	0	0
08:05	5	17.32	288.21	3.84	0.154
08:10	10	_	_	_	_

It is recommended that as soon as the water absorption slows down, the data obtained be plotted to determine whether any anomalies have resulted. In most cases, anomalies are due to errors in the weighing or in the time measurement.

After the sample has reached the asymptotic water absorption value, leave the sample 24 hours and then take the last weighing ( $W_{\rm asymp}$ ). The capillary water absorption curve plots  $M_{\rm i}$  versus square root of time (see Figure 6.2 in "Results and Data Presentation").

#### **Total Water Immersion**

As soon as the last weight in the capillary water absorption test is obtained, completely immerse the sample in a glass beaker or other suitable container of de-ionized water; it should be left immersed for 24 hours. Then remove the sample, softly pat it dry, weigh it  $(W_{\rm max})$ , and note the time. This is the first point of the drying curve and will be used to calculate the total porosity accessible to water at room pressure.

#### **Evaporation Curve**

The easiest procedure is to leave the sample on the balance for at least the first 15 minutes to half hour, because evaporation of the water is fast. Weight should be recorded regularly, noting the corresponding time. Again, a six-column table (Table 6.3) should be prepared to record actual time of measurement, cumulative time in both minutes and hours, weight of the sample, moisture content, and moisture content per unit volume ( $U_t$ /volume of sample; g/cm³). The drying curve plots moisture content on the *y*-axis as a function of time in hours.

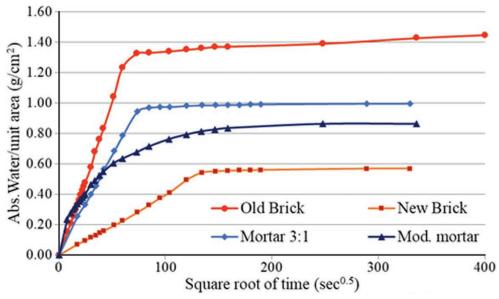
**TABLE 6.3.** Evaporation curve test data for drying of sample 2 (an example). Moisture content is calculated per unit volume of the sample (125 cm $^3$ );  $U_{\rm t}$  = amount of water absorbed (change in weight); a dash (—) = no measurement or calculation made.

Actual time	Cumula	tive time	Weight at time	Moisture content	Moisture content per volume
(hr)	(min)	(h)	$(W_{\rm t})$ (g)	( <i>U</i> <sub>t</sub> ) (g)	$(U_{\rm t}/{\rm vol.})$ (g/cm <sup>3</sup> )
08:00	0	0	254.52	39.93	0.31944
08:01	1	0.017	254.49	39.90	0.31920
08:02	2	0.033	254.46	39.87	0.31896
08:03	3	_	_	_	_

#### **Results and Data Presentation**

For illustration purposes, Figure 6.2 shows the capillary absorption curves for two bricks, a traditional handmade one and a new machine-made version, as well as two mortars, one made of a natural hydraulic lime: sand (1:3) mixture and the other a "modified" mortar made from white Portland cement: lime putty: sand (1:2:9) with the addition of a 10% acrylic emulsion.

The initial straight section of the curves (the slope of the ascending line) corresponds to the capillary water absorption coefficient ( $g/m^2$ .sec<sup>0.5</sup>). The acrylic emulsion changes significantly the absorption pattern of the mortar. The data are presented in Table 6.4.



**FIGURE 6.2.** Capillary absorption curves (amount of water absorbed per unit area,  $M_i$ ) for the handmade (old) and the machine-made (new) bricks, the 1:3 natural hydraulic lime mortar, and a modified Portland cement with lime mortar and an acrylic emulsion.

**TABLE 6.4.** Capillary water absorption coefficient (CAC) and the correlation factor<sup>a</sup> that serves to evaluate the how straight the line is (a straight line has a correlation factor of 1); the length of time used to calculate the CAC; the asymptotic water content; and approximate time to reach it.

	CA	С	_		Asymptotic		
Sample	(g/cm². sec <sup>0.5</sup> )	(kg/m². h <sup>0.5</sup> )	Length of time (h)	Correlation factor a	water content (g/cm²)	Approx. time (h)	
Old brick	0.00206	1.2	4	0.9994	~1.50	40	
New brick	0.00404	2.4	4	0.9985	0.57	10	
Mortar	0.01277	7.7	1.5	0.9994	1.00	10	
Modified mortar	0.01453	8.7	0.16	0.9486	0.86	30	

<sup>&</sup>lt;sup>a</sup>The correlation factor serves to indicate how straight the line is, its value is obtained when performing the plot in Excel.

The capillary absorption coefficient for the old brick was twice that for the new brick, indicating that it absorbed water twice as fast, reflecting an overall greater number of larger pores in the capillary range (Table 6.1), while the asymptotic capillary absorption value was about three times higher, indicating an overall larger porosity.

The mortars, on the other hand, had more similar values for the capillary water absorption coefficient, and they fell within the values obtained for the old brick. This was confirmed by the total immersion test that serves to determine water absorption (%), formerly called apparent porosity (ASTM C67-00, 2000; ASTM C97/C97M-09, 2009), water absorption capacity ( $[W_{\rm max} - W_{\rm dry}] \times 100/W_{\rm dry}$ ) (Borrelli, 1999), imbibition capacity( $[W_{\rm max} - W_{\rm dry}]/W_{\rm dry}$ ), and open porosity percentage. The last is calculated by the ratio of the volume of open pores ( $V_{\rm op}$ ) to the total volume of the sample ( $V_{\rm S}$ ). For practical purposes it can be calculated from the total amount of absorbed water divided by the density of water, ( $W_{\rm max} - W_{\rm dry}$ )/d, where density (d) is taken to be 1 g/cm³, the value it has at 4°C. The results are shown in Table 6.5.

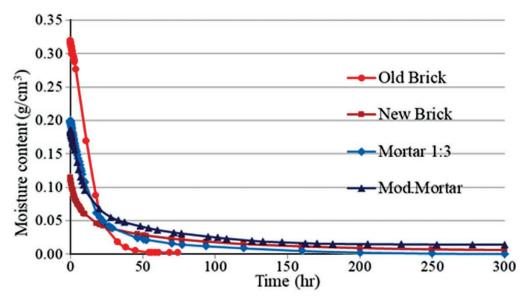
**TABLE 6.5.** Water absorption capacity (WAC, weight per weight [wt./wt.]; % = [ $W_{\rm max} - W_{\rm dry}$ ] × 100/ $W_{\rm dry}$ ), imbibition capacity (IC, wt./wt.; [ $W_{\rm max} - W_{\rm dry}$ ]/ $W_{\rm dry}$ ), and open porosity (vol./vol.; % = [ $W_{\rm max} - W_{\rm dry}$ ] × 100/d) for the four materials tested, where  $W_{\rm max}$  = saturated weight;  $W_{\rm dry}$  = dry (initial) weight; and d = density of water at 4°C.

	WAC		Open porosity
Sample	(%, wt./wt.)	IC (wt./wt.)	(%, vol./vol.)
Old brick	18.61	0.186	32
New brick	4.50	0.045	11
Mortar	9.80	0.098	20
Modified mortar	9.15	0.091	18

As previously estimated, the open porosity of the old brick was about three times that for the new brick, and both mortars had an intermediate open porosity. It is important to remember that the open porosity is not the total porosity of the material. This can only be obtained if the sample is saturated with water under vacuum. However, for practical purposes, open porosity, which is the porosity accessible to water at atmospheric pressure, is sufficient since building materials would seldom be totally saturated with water.

The evaporation curves obtained for the moisture content (g/cm<sup>3</sup>) as a function of time (h) following the total immersion are shown in Figure 6.3.

The curves show that although the handmade brick absorbed significantly more water than the two mortars and the new brick, it still dried first, confirming the larger range of capillary pores. Within two days it was practically dry. The 1:3 natural hydraulic lime mortar and the new machine-made brick took more than a week to dry, and the mortar with the acrylic emulsion required nearly two weeks to dry. For materials to be compatible, their water absorption, and especially their drying characteristics, must be matched, as otherwise deterioration will occur preferentially on the material that retains moisture longer. The 1:3 natural hydraulic lime mortar was shown to be compatible with the machine-made brick, whereas the handmade brick would require a more porous mortar. In general, organic additives, while useful for consolidation, have the disadvantage of taking far longer to dry.



**FIGURE 6.3.** Evaporation curves for handmade (old) bricks and machine-made (new) bricks, the 1:3 natural hydraulic lime mortar, and a modified Portland cement with lime mortar and acrylic emulsion.

From the linear section of the curves, the initial and final drying rates can be calculated as shown in Table 6.6. The correlation factors (obtained via Excel from the straight portions of the curve) ensure that the rates correspond to the section of each curve that is as straight as possible. From practical experience, these factors should be >0.995 for the initial drying rate and >0.95 for the ending drying rate, since the weighing errors that can occur as the sample attains its dry weight are larger.

**TABLE 6.6.** Initial and final drying rates for the four materials, length of time for which they are valid, and the corresponding correlation factors<sup>a</sup>, as well as the residual moisture content and the days required to reach it.

Sample	Initial drying rate (g/cm³.h)	Time (h)	Correl.	Final drying rate (g/cm³.h)	Time (h)	Correl.	Residual moisture content (g/cm³)	Time (days)
Old brick	-0.013	17	0.998	-1.52 E-05	18	0.946	0.002	3
New brick	-0.010	3	0.995	-7.47 E-05	230	0.953	0.006	12
Mortar	-0.010	10	0.995	-6.35 E-05	220	0.946	0.001	10
Modified mortar	-0.009	10	0.997	-1.18 E-05	230	0.958	0.015	16

<sup>&</sup>lt;sup>a</sup>The correlation factors are calculated via Excel from the two straight sections of the curve—the initial portion and the final one.

# **Concluding Remarks**

The water is absorbed by capillary pores within a few hours, especially if these pores fall within the larger range (diameters from 10  $\mu$ m to about 1 mm), as is the case for the handmade brick. However, if the overall porosity is higher, it may take more than a day of continuous wetting to achieve constant water content. On the other hand, the drying takes nearly three times longer than wetting for this type of brick, whereas for the machine-made brick drying takes 24 times longer, as it has far more fine capillary pores (diameters of about 0.1–10  $\mu$ m), which tend to retain moisture; this is also the case for the 1:3 mortar. Drying would take far longer for the case of the modified mortar, since the moisture content reached by the time the experiment was ended was 10 times than that of the 1:3 mortar. This moisture content can be attributed to the formulation that contains Portland cement and an acrylic emulsion.

# APPENDIX: BIBLIOGRAPHY OF SUGGESTED RESOURCES AND FURTHER READING

#### A. ELENA CHAROLA

A bibliography of suggested publications, many of which can be found online, is provided here to aid readers in identification of potential problems and their solutions. It includes other manuals and publications that provide important information on the deterioration and conservation of building materials. In particular, the *ARC Laboratory Handbook* prepared by Borrelli and Urland (1998–1999) presents useful information, tests, and protocols under the sections on porosity, salt, binders, and color specification and measurement. The bibliography is organized by topic, such as brick, stone, and so on. From the section "Compatibility Assessment for Conservation Actions," all four publications are recommended to provide guidance for problem solving in conservation interventions. Problem solving is a process that requires understanding the complexities involved in the deterioration of materials, as well as in matching solutions to the resources available.

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