# Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces

## A Manual of Laboratory and Field Test Methods

Beril Biçer-Şimşir
and
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The Getty Conservation Institute works internationally to advance conservation practice in the visual arts—broadly interpreted to include objects, collections, architecture, and sites. The GCI serves the conservation community through scientific research, education and training, model field projects, and the dissemination of the results of both its own work and the work of others in the field. In all its endeavors, the GCI focuses on the creation and delivery of knowledge that will benefit the professionals and organizations responsible for the conservation of the world's cultural heritage.

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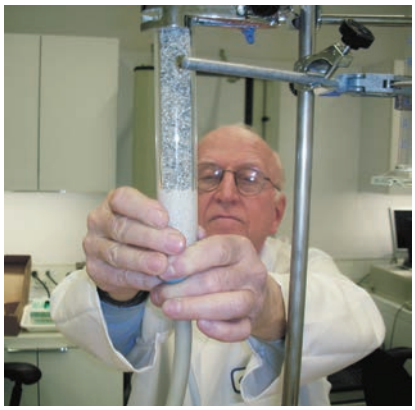

*In memory of Professor Giorgio Torraca (1927–2010), whose pioneering work on the development and testing of injection grouts formed the basis of the Getty Conservation Institute project on their testing and evaluation; his guidance and mentoring greatly helped to shape this manual.*

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**Part II Field Testing Procedures**

# Foreword

From 1980 to 1984, a team of scientists and conservators, led by Professor Giorgio Torraca at the International Centre for the Study of the Preservation and Restoration of Cultural Property (ICCROM), worked together to carry out in-depth testing of injection grouts for architectural surfaces, primarily historic plasters, wall paintings, and mosaics. This research led to the development of tests and grouting formulations that have since been used extensively in the conservation field. The work greatly influenced conservation practice, providing a much-needed solution that permitted the reattachment of significant decorated surfaces in situ.

In the last two decades, a number of commercial grouts have entered the market, and they are widely used by conservators around the world as an alternative or a complement to custom formulations. In order to help conservators make informed choices regarding the various grouts that have become available since ICCROM's seminal research, the Getty Conservation Institute (GCI) recently undertook a project to evaluate the range of custom-mixed and commercially available injection grouts in widespread use. Throughout the project, the GCI was fortunate to have the strong support of Professor Torraca, who advised on research design and assisted in the interpretation of results. At the GCI, the work was carried out by a scientist-conservator team led by Beril Biçer-Şimşir, assistant scientist in building materials, and Leslie Rainer, senior project specialist and wall paintings conservator.

A preliminary literature review and investigation of injection grouts available in the field indicated that, while a wide range of grouts has been developed for diverse applications, there is little consistency in testing procedures, and no standard test methods have been specifically developed for this class of grouts. Instead, researchers and manufacturers have turned to a number of standard tests for other materials, including mortars, plasters, concrete, and structural grouts. Although injection grouts share some properties with these related materials, the parameters for testing and evaluation are often different, and tests are typically modified to accommodate different particle size, strength, shrinkage, and other factors. Thus, results from tests carried out by different companies or different researchers are seldom comparable.

To address this issue, the GCI team has developed a suite of laboratory and field test procedures that can be used by scientists and conservators specifically to evaluate injection grouts for the conservation of plasters, wall paintings, and mosaics. The laboratory tests are based on standard procedures used in allied fields for similar materials, but they have been modified to correspond to the properties and performance parameters primarily of lime-based hydraulic grouts. Field tests are loosely based on the laboratory tests, using simplified procedures and equipment that is more likely to be available to conservators in the field. The full suite of tests permits a comprehensive evaluation of grout properties and performance when new grouts are developed, existing mixes are substantially modified, different grout formulations are compared, and individual properties for specific applications and field conditions are verified.

This volume is one of many GCI publications aimed at disseminating the results of our research to the conservation community. It is our hope that, by providing scientists and conservators with reliable and comparable laboratory and field tests for the evaluation of injection grouts, this publication will advance conservation practice and lead to more informed conservation decisions.

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The authors would like to acknowledge the many colleagues who helped shape this work and who contributed to it at different stages of the project. First, we would like to extend our heartfelt gratitude to Professor Giorgio Torraca for his unfaltering support of this project from its inception to his last review of the draft document. Without his guidance and mentoring, this project and these tests would not have come together in this manual. At the GCI, Jeanne Marie Teutonico, associate director, programs, and Giacomo Chiari, chief scientist, have given us full support of the research. David Carson, a close colleague, has helped solve practical issues of instrumentation setup and testing development and has given us all-around good advice. For the development of the ion chromatography procedure, the combined efforts of Mara Schiro, Joy Mazurek, and Cecily Grzywacz are greatly appreciated. We are grateful to Isobel Griffin and Benedicte Bertholon-Palazzo, who helped draft the preliminary literature review and compile the bibliography, which allowed us to identify gaps in the research and to develop appropriate test methods based on standard tests for similar materials. Hande Gunozu was responsible for preliminary development of the field tests and provided valuable assistance in the laboratory. Traci Lucero likewise assisted in the laboratory as tests were developed. Stacey Rain Strickler of the J. Paul Getty Museum Imaging Services and Holly Brobst advised on photography for images used throughout this manual. Elsa Bourguignon, William Ginell, and John Fidler were of tremendous help as external reviewers of the manuscript. Finally, we would like to acknowledge Angela Escobar, who shepherded this volume through the editing and design process, and designer Gary Hespenheide. Together they helped turn these testing procedures into a manual for conservation scientists and conservators to use in the laboratory and field.

# Abbreviations

| | |
|---|---|
| $\dot{\gamma}$ | shear rate ($s^{-1}$) |
| $\delta$ | thickness (mm) |
| $\eta_{pl}$ | plastic viscosity (Pa · s) |
| $\rho_{dry}$ | dry density ($g \cdot cm^{-3}$) |
| $\rho_{wet}$ | wet density ($g \cdot cm^{-3}$) |
| $\sigma$ | shear stress (Pa) |
| $\sigma_B^*$ | apparent Bingham yield stress (Pa) |
| $\omega$ | water to grout ratio by weight |

| | |
|---|---|
| a | area ($m^2$) |
| A | water absorption coefficient ($kg \cdot m^{-2} \cdot s^{-1/2}$) |
| $A_{field}$ | water absorption coefficient measured in the field ($kg \cdot m^{-2} \cdot s^{-1/2}$) |
| B | bleeding (%) |
| CE | combined expansion (%) |
| d | diameter (mm) |
| $d_{max}$ | maximum aggregate size (mm) |
| $\Delta M$ | weight change (g or %) |
| $\Delta P$ | vapor pressure difference (Pa) |
| E | expansion of grout (%) |
| f | splitting tensile strength ($N \cdot mm^{-2}$) |
| func( ) | function |
| F | breaking load (N) |
| $f_{sb}$ | shear bond strength ($N \cdot mm^{-2}$) |
| FB | final bleed water (%) |
| h | height of the grout in the column (mm) |
| l | length (mm) |
| L | distance (mm) |
| m | weight of water absorbed per unit area ($kg \cdot m^{-2}$) |
| M | weight (g or kg) |
| r | radius (mm) |
| $R^2$ | coefficient of determination |
| RH | relative humidity (%) |
| S | drying shrinkage (%) |
| $S_{vp}$ | saturation vapor pressure (Pa) |
| t | time (s or h) |
| $T_i$ | thixotropy index |
| V | volume (mL) |
| w | width (mm) |
| W | water content (g or %) |
| WRV | water retention value (%) |
| WVP | water vapor permeability ($g \cdot h^{-1} \cdot Pa^{-1} \cdot m^{-1}$) |
| WVTR | rate of water vapor transmission ($g \cdot h^{-1} \cdot m^{-2}$) |
| $WVTR_{field}$ | rate of water vapor transmission measured in the field ($g \cdot h^{-1} \cdot m^{-2}$) |

# 1. Introduction



This manual provides a set of suggested procedures for the testing of lime-based hydraulic injection grouts for the conservation of architectural surfaces. It is meant to be used as a reference by conservation scientists in the laboratory and by conservators working in the field to test, evaluate, and select appropriate injection grouts for the conservation of delaminated wall paintings, plasters, and mosaics on vertical and horizontal surfaces. It is important to note that these test procedures have not been designed for structural grouts, which have different properties and performance requirements; rather, they have been specifically designed for injection grouts that are used to fill voids and reattach delaminated plasters, wall paintings, and mosaics and that do not perform a structural function.

The information presented here is based on the results of a research project, initiated in 2005, that the Getty Conservation Institute (GCI) has undertaken on this topic. As part of this project, following a related literature review,[1] this suite of laboratory and field testing procedures was developed to evaluate injection grouts specifically for architectural surfaces. In this manual, the procedures are presented in two parts—part I, Laboratory Testing Procedures, and part II, Field Testing Procedures.

The laboratory testing procedures presented here have been developed for use by scientists, conservation scientists, and conservators working in research or university laboratories that are equipped for materials testing.[2] They can also be used for reference by practicing conservators, architects, and engineers who may be commissioning testing and who wish to become familiar with testing procedures that a laboratory can provide. Most of the laboratory testing procedures are based on standard test methods (e.g., EN, ASTM, UNI, etc.; see appendix A for a list of standards) developed for other materials (e.g., mortars, epoxy resins, hydraulic or cement binders, etc.) that are not fully representative of the properties and performance of injection grouts used for the conservation of architectural surfaces. For each procedure, the most suitable standard test method or methods were identified, modified, and adapted as necessary to evaluate lime-based hydraulic injection grouts, including natural hydraulic lime-based grouts and lime-pozzolan grouts.[3] Procedures have been divided into qualifying tests, those needed when formulating or substantially modifying a grout; and supplementary tests, which are useful for grouts that require specific working properties and performance characteristics.

The simple field tests have been designed for use by conservators and do not require specialized instrumentation or full laboratory setups. The tests are mainly useful for comparing grouts and for quality control. They loosely follow the laboratory test procedures described above and can be used to validate the results of prior laboratory tests, which have been performed under controlled environmental conditions.

Some of the test procedures provided in this manual can be used without modification for testing nonhydraulic injection grouts, including earth or lime grouts.[4] These are identified in the relevant test procedures. For other test procedures, modifications such as sample preparation, curing time and condition, and so on must be made when evaluating nonhydraulic injection grouts. All the tests are developed and used at sea level. Additional considerations may be required when they are used at higher elevations.

While it may be necessary only at times to carry out the full suite of tests, in many cases, certain properties may need to be verified, and then individual tests may be used separately or in combination. For this reason, all steps are presented for each test procedure.

It is hoped that this manual proves to be useful in the laboratory and in the field as a reference source that includes a comprehensive set of laboratory and field testing procedures specifically developed to address the properties and parameters of lime-based hydraulic injection grouts for the conservation of architectural surfaces.

This manual does not include health and safety warnings regarding the use of chemicals, grout materials, and laboratory equipment. The users of this manual should establish appropriate health and safety practices.

Notes

1. B. Biçer-Şimşir, I. Griffin, B. Palazzo-Bertholon, and L. Rainer, Lime-based injection grouts for the conservation of architectural surfaces, *Reviews in Conservation* 10 (2009): 3–17.
2. A list of equipment needed to carry out the laboratory procedures is presented in appendix B.
3. Lime-based hydraulic injection grouts were selected because of their compatibility with historic substrates in terms of their physical and chemical properties, mechanical strength, porosity, permeability, and other factors. Commercially produced grouts for conservation and custom-mixed grouts formulated by conservators are also most frequently of lime-pozzolan or hydraulic lime-based composition.
4. It should be noted that nonhydraulic grouts require drying and access to carbon dioxide (i.e., carbonation for lime-based grouts) to gain rigidity and are less frequently used as injection grouts because of slow setting when used behind an architectural surface to fill voids or reattach a plaster, wall painting, or mosaic.

## 1.1. Definition

An injection grout for the conservation of architectural surfaces is a bulked fluid material that can be injected behind plaster, wall paintings, or mosaics to fill cracks and voids and reestablish adhesion between delaminated layers upon setting. Injection grouts are composed of one or more binders, aggregates, admixtures, and fluid, typically water.

## 1.2. Injection Grouting Practice

Injection grouting is a method commonly used by conservators for filling voids and cracks and for reattaching plasters, wall paintings, and mosaics to architectural substrates. Injection grouting differs from structural grouting in the scale of implementation. More significantly, as previously noted, it does not perform a structural function. Injection grouting for the conservation of architectural surfaces involves several steps, described below.

### Preparation of the Substrate Prior to Injecting the Grout

Preparation of the substrate (wall, ceiling, vault, floor, etc.) is critical to a successful grouting intervention. Debris, which has accumulated in voids behind the wall, should be thoroughly cleaned out. This may be done by aspiration, using mini vacuum attachments (e.g., computer vacuum attachments), air bulbs, and other small tools that can empty cavities of debris. Often a small hole must be made in the architectural surface material to empty debris. Water or water and alcohol may also be used to flush the cavity. Similarly, interior surfaces can be prewetted to facilitate passage of the injection grout through interior networks, to ensure good adhesion of the grout, and to avoid rapid drying. When liquids are used to flush or prewet, caution should be taken not to leave excess water in the void behind the surface, as it can dilute the grout mixture and change its properties. In addition to this, water used to flush or prewet can mobilize the existing salts and lead to efflorescence. Plant growth should be removed prior to grouting, particularly in the case of floor mosaics, as root systems might also prevent the grout from flowing properly.

In some instances, small airholes may be drilled to facilitate passage of the grout. However, on most wall paintings, this is not advised, and typically, preexisting holes, cracks, or losses are used as injection ports when possible. For large voids, some architectural conservators drill a network of application holes, which may be done on plain plaster surfaces, but this is clearly not desirable for wall paintings conservation. Holes that the grout could leak through should be sealed to prevent grout from dripping onto the surface; fine cracks may be faced, and if necessary, the edges of the area to be treated can be sealed with materials such as plasticine or cyclododecane, or with a mortar used for fills.

### Injecting Grout through a Syringe or Larger Tube

Once the architectural surface and substrate have been prepared, the grout mixture is introduced behind the plaster, wall painting, or mosaic surface. The technique utilized to introduce grout into the void depends upon the size, accessibility, and alignment of the void. If the void can be accessed from the top, then a fairly liquid grout can be introduced at the upper edge and allowed to flow down inside the void, as gravity grouting, which can also be used for floor mosaics. Otherwise, grout is usually injected by syringe with a cannula of appropriate size. Results of this research show that for the majority of grout mixtures, a 12 or 14 gauge cannula can be used to inject most injection grouts. For especially large voids, transfusion tubing can also be used. When a particularly large cavity is grouted, application should occur in stages, to give each grout application sufficient time to lose water and become fairly dry and hard.

Frequently, and particularly in the case of fragile architectural surfaces and vaults, it may be necessary to support the plaster, wall painting, or mosaic in place by the use of supports while grouting is being carried out and during setting. Applying pressure to the grouted area can, in some cases, also realign a deformed plaster and provide better contact and adhesion.

#### Cleanup of the Surface

Following grouting, the surface should always be checked to ensure that no grout remains on the surface. Grout may flow and reappear on the surface in unexpected places. During grouting, work should be monitored, and cleanup should be done immediately. Grout left on the surface is likely to set and may discolor the surface on which it is deposited if it is not removed in a timely fashion.

## 1.3. Working Properties and Performance Characteristics of Injection Grouts

To ensure the optimum performance of commercial and custom-mixed injection grouts, a wide range of working properties and performance characteristics needs to be evaluated. A minimum number of properties should be tested to determine the behavior of a grout in the initial wet state, during setting, and after curing. Commercially available grouts may have been optimized for fundamental parameters, such as injectability, flow, and shrinkage. However, specific properties may require testing to ensure that the appropriate grout is being used for a specific purpose. When custom-mixed grouts are developed, the full range of properties should be tested first in the laboratory. For specialized cases that require grouts with specific properties, supplementary tests may be required. For both commercially available and custom-mixed grouts, results of the laboratory testing should be validated by testing in the field. Injection grouts should meet the following performance criteria:

- Grouts should be compatible with the original substrate and surface materials.
- Grouts should be fluid enough to be injected with no clogging.
- Grouts should have minimal separation of components.
- The volume shrinkage of a grout (from wet paste to hardened solid) should be minimal.
- The concentration of soluble salts in the grout should be as low as possible.
- The mechanical strength (i.e., compressive, shear, or tensile strength) of the cured injection grout should be similar to, but less than, the original plaster and substrate to avoid creating excessive stresses on the original material.
- The capillary water absorption of the cured injection grout should be similar to that of the original material.
- The cured grout should allow the passage of water vapor corresponding to the water vapor permeability of the original materials and should not create a vapor barrier.
- Grouts should provide an adequate bond, and the bond strength at the interfaces should be similar to but less than the strength of the original materials.

Three categories of tests have been identified and presented in this manual:

- qualifying laboratory tests that determine the critical parameters for acceptable grout performance for general applications
- supplementary laboratory tests that should be performed to evaluate grouts for specific uses, such as very liquid grouts for filling fine cracks, lightweight grouts, etc.
- field tests that can be used on site for comparison of different injection grouts that have been previously tested in the laboratory, and for quality control of materials and grout mixtures

Additionally, a number of laboratory analytical tests can be performed to evaluate the chemical and physical characteristics of grouts or individual grout components. These include:

- elemental analysis (e.g., X-ray fluorescence [XRF] and scanning electron microscopy with energy dispersive X-ray analysis [SEM-EDX])
- compositional analysis (e.g., X-ray diffraction [XRD], thermogravimetric analysis–mass spectrometry [TGA-MS], and petrography)
- organic component content (e.g., Fourier transform infrared spectroscopy [FTIR])
- particle size distribution (sieve analysis and laser diffractometry)
- true density or specific gravity (helium pycnometry)

These tests may also help to determine the intentional or unintentional material changes that can occur over time in commercial grout formulations and

ingredients used in custom-mixed grouts. Quality control of the ingredients of injection grouts becomes important, especially when custom-mixed injection grouts are used, since the use of high-quality materials is crucial for obtaining consistent, effective, and high-quality grouts. However, a description of these analytical methods is not included in this manual, since chemical and physical properties of injection grouts cannot always be directly correlated with working and performance properties, and interpretation of the results of material analysis, especially for commercial grouts that contain numerous admixtures (e.g., plasticizers, antiflocculants, biocides, etc.), is not straightforward. Experience in the GCI research project showed that these tests proved most helpful and relevant when specific questions about materials arose.

Another group of test methods not included in this manual are durability tests such as freeze and thaw resistance and resistance to soluble salts. While these tests can provide information on the performance of concrete, bricks, and mortars, this information is only relative. For injection grouts, it is even more difficult to relate durability test results directly to performance in practice, mainly because grouts are injected into composite systems and are not directly exposed to these conditions.

## 1.4. Test Methods

### 1.4.1. Qualifying Laboratory Tests for Injection Grouts

These test methods (table 1.4.1) are grouped as qualifying test methods since they determine the properties of

**Table 1.4.1** Qualifying laboratory tests for the evaluation of injection grouts.

| Working Properties | | | |
|---|---|---|---|
| **Laboratory test** | **Test method** | **Standard ref.** | **Section** |
| Injectability[1] | The grout is injected at constant pressure from the bottom of a vertically held column (transparent plastic tube) filled with granular material. Injectability of grout in a capillary network under predefined pressure is defined by the distance traveled by the grout as a function of time. | EN 1771 (2005) | 2.1 |
| Expansion and bleeding | Freshly mixed grout is placed in a graduated cylinder, and the accumulation of bleed water and amount of expansion are measured as a function of time. | ASTM C 940-03 | 2.2 |
| Wet density[2] | Grout density is determined by weight in a known-volume container. | ASTM C 185-02, sect. 9.4 | 2.3 |
| **Properties during Setting and Curing** | | | |
| **Laboratory test** | **Test method** | **Standard ref.** | **Section** |
| Drying shrinkage | The final volume of the cured grout sample is calculated using the weights of the sample in a displacement fluid (i.e., kerosene) and in air. | ASTM C 474-05 | 2.4 |
| **Hardened Properties** | | | |
| **Laboratory test** | **Test method** | **Standard ref.** | **Section** |
| Splitting tensile strength | A cylindrical hardened grout specimen is tested on its side in diametral compression. | EN 1771 (2004) | 2.5 |
| Soluble salt content by ion chromatography | The amount of water-soluble salts extracted from hardened grout is obtained by ion chromatography. | ASTM D 4327-03 | 2.6 |
| Capillary water absorption | Water absorption of hardened grout is measured by a gravimetric method. | NORMAL 11/85<br>RILEM test no. II.6<br>EN 1771 (2005) | 2.7 |
| Water vapor transmission by the wet cup method | The rate of vapor movement is determined by the weight loss, over time, of a cup partially filled with water, blocked and sealed with a grout specimen, under controlled conditions. | NORMAL 21/85<br>ASTM E96-05 | 2.8 |

[1] This test should be used to determine the injectability of all custom-mixed grouts and any grout needed to fulfill specific conditions, such as fine-crack grouting, highly absorptive media, limited or no prewetting, and so on.

[2] This test can be used to identify the lightweight grouts when the addition of weight to a structure or surface layer due to grouting is considered to be important.

grouts that are crucial for their performance and compatibility within the architectural system. Qualifying laboratory tests are divided into three groups: tests determining working properties (2.1–2.3), properties during setting and curing (2.4), and hardened properties (2.5–2.8).

### 1.4.2. Supplementary Laboratory Tests for Specific-Application Grouts

These test methods (table 1.4.2) are used for grouts that are needed for specific applications such as the grouting of fine cracks (microgrouting), deep voids, large detached areas, vaults and ceilings, and so on; supplementary laboratory tests should be conducted to assess the performance of the grout under such conditions. Supplementary laboratory tests are also divided into three subgroups: working properties (3.1–3.3), properties during setting and curing (3.4), and hardened properties (3.5).

### 1.4.3. Field Tests

The field tests described in this manual have been developed at the GCI for the purpose of on-site testing of injection grouts. These tests, which are derived from the laboratory tests, take into account practical considerations of working on site, where laboratory facilities and instrumentation may not be available. Properties that can be tested in the field relate mainly to the workability of the grout in the wet state and to properties of the grout during setting. Only a few performance characteristics—such as water absorption and water vapor transmission—may be easily



**Table 1.4.2** Supplementary laboratory tests to evaluate injection grouts for specific applications.

| Working Properties | | | | |
|---|---|---|---|---|
| **Laboratory test** | **Test method** | **Standard ref.** | **Suggestions** | **Section** |
| Flow with injectability columns | The grout is poured into a vertically held column (transparent plastic tube) filled with granular material. Injectability of grout into a capillary network under gravity is defined as the distance traveled by the grout as a function of time. | EN 1771 (2005) | This test should be used when gravity grouting will be conducted. | 3.1 |
| Water retention and release | Grout is contained in a perforated dish covered with a filter paper and exposed to suction for 1 minute. | DIN 18 555-7 (1987)<br>ASTM C 1506-03 | If the injectability test cannot be performed, this test may provide information about flow and injectability, as the higher the water retention capacity of a grout, the farther it will flow. | 3.2 |
| Rheological measurements | The flow resistance of a grout is determined using a commercial rotational viscometer. | UNI 11152-05 | Viscosity measurements are useful when designing a new grout with a specific flow behavior or when comparing viscosities of different grouts for a specific use. | 3.3 |
| **Properties during Setting and Curing** | | | | |
| **Laboratory test** | **Test method** | **Standard ref.** | **Suggestions** | **Section** |
| Time of setting by Vicat needle | The time of setting is determined by periodically inserting a needle into grout and measuring the depth of penetration of Vicat needle. | ASTM C 953-06<br>ASTM C 191-08 | This test is suggested when the length of time needed for the grout to set is important (e.g., if the plaster is soft or fragile) to estimate time needed before supports can be removed. | 3.4 |
| **Hardened Properties** | | | | |
| **Laboratory test** | **Test method** | **Standard ref.** | **Suggestions** | **Section** |
| Shear bond strength | Stone couplets are adhered with grout and tested under shear. | ASTM D 905-08<br>EN 196-1 (2005) | This test is suggested when bond strength plays an important role (e.g., adhering large detached areas). | 3.5 |

**Table 1.4.3** Field tests developed for injection grouts.

| Working Properties | | |
|---|---|---|
| **Field test name** | **Test method** | **Section** |
| Injectability with syringe | Grout is poured into a vertically held syringe that is partially filled with granular material, and pressure is applied on the grout with the plunger. | 4.1 |
| Flow with syringe | Grout is poured into a vertically held syringe that is partially filled with granular material, and the penetration of grout into the capillary network is observed. | 4.2 |
| Flow on plastered tile | A constant volume of grout is injected into a vertical crevice in plaster applied to a vertically placed tile, and the distance it flows is determined. | 4.3 |
| Expansion and bleeding | Grout is placed in a graduated cylinder, and accumulation of bleed water and amount of expansion are measured. | 4.4 |
| Wet density | Grout is weighed in a syringe, and the density is calculated. | 4.5 |
| **Properties during Setting and Curing** | | |
| **Field test name** | **Test method** | **Section** |
| Drying shrinkage | Dimensional changes, including cracks, of a grout specimen placed in a plastic or mortar cup are observed. | 4.6 |
| Final setting time | The time of setting is determined by periodic insertion of a cannula into a cup filled with grout until solidification occurs. | 4.7 |
| **Hardened Properties** | | |
| **Field test name** | **Test method** | **Section** |
| Capillary water absorption | Water absorption of a hardened grout specimen is measured by a gravimetric method, following the procedure for the laboratory test. | 4.8 |
| Water vapor transmission rate by the wet cup method | The rate of water vapor transmission through a cured grout sample is determined gravimetrically under field conditions. This test follows the procedures for the laboratory test. | 4.9 |

tested in the field. Field tests for adhesion are not included since they require special setups, they are not always reproducible with reliable results, and the interpretation of their results is not straightforward. Additionally, testing on the original substrate is often not possible. The list of field tests developed for injection grouts is given in table 1.4.3. Field tests are also divided into three groups: working properties (4.1–4.5), properties during setting and curing (4.6 and 4.7), and hardened properties (4.8 and 4.9).

In addition to serving the purposes of comparison of grouts and quality control, field tests may also provide useful feedback for additional laboratory testing of modifications to injection grouts for specific sites or conditions and provide useful baseline data and information for communication between field and laboratory. Field tests can help overcome issues of grout performance by providing information on the origin of the problem (e.g., injectability, shrinkage, etc.), by providing information on changes at the site that may have led to the performance problem (e.g., environmental conditions, materials, preparation process, etc.), and by enabling identification of modifications that may help to solve the problems.

While field tests can be very valuable, their limitations should also be recognized. Field tests are not as precise as laboratory tests and cannot be used to design new grouts or substantially modify grouts without sufficient laboratory testing. Their primary purpose is as a tool for comparison of grouts and for quality control. It is important to know that the results obtained may have a high coefficient of variation and are rarely reproducible, mainly due to fluctuating environmental conditions and varying substrates that cannot be controlled as in a laboratory setting.

New grouts or substantial modifications of grouts should first be tested in the laboratory to ensure that their properties and performance are appropriate for use on site. Laboratory testing should be followed up by field testing, and final modifications should be tested in the laboratory and the field to ensure optimal performance of the grout on site.

## 1.5. Preparation of Injection Grouts

The preparation procedure for injection grouts is important since it affects their workability and their final performance characteristics. Both the stirring method and the time of mixing have a vital influence on the consistency of the grout mixture. In general, an injection grout mixed at high speed for a longer time produces a grout with better injectability and penetration and less separation of liquid and solid phases.[1] In the field, an adjustable-speed mixer similar to those used in the laboratory is suggested, although a variable-speed power drill with an impeller, a milkshake mixer, or a soup mixer can also be used. It is important to prepare the grout at the site following the same procedure used in the laboratory.

When a commercial grout is used, the manufacturer's suggestions on preparation, including stirring technique, water content, and time of mixing, should be followed. When a custom-mixed grout is used, the grout ingredients should be measured accurately, and the mix proportions should be followed. The optimum stirring technique and time of mixing should be determined and used in the laboratory and in the field.

If feasible, it is strongly suggested that volume ratios be converted into weight ratios to minimize measurement errors. The amount of water used for injection grouts is an important factor that can modify flow, shrinkage, and bonding. In order to obtain consistent grout batches, the amount of water should be accurately measured at the time of mixing to avoid evaporation, and the water to grout weight ratio should be kept constant for each mixing. An example of the mixing procedure used to prepare injection grouts at the GCI is as follows:

1. Pour premeasured water and, if applicable, liquid additives into the mixing bowl.
2. Add premixed dry ingredients within 30 seconds while mixing at low speed (200–300 rpm) using a universal variable-speed stirrer (e.g., Caframo BDC 3030 Stirrer; see fig. 1.5.1).
3. Increase speed to 1000–2000 rpm, depending on the grout, and continue mixing for at least 4 minutes.
4. Following mixing, pass the fresh grout through a 1 mm sieve to remove any clumps and to improve ease of injection.

### Note

1. K. Zajadacz and S. Simon, Grouting of architectural surfaces: The challenge of testing, in *Theory and Practice in Conservation: International Seminar,* ed. J. Delgado Rodrigues and J. Mimoso (Lisbon: National Laboratory of Civil Engineering, 2006), 509–17; and A. Miltiadou-Ferons, Criteria for the design of hydraulic grouts injectable into fine cracks, and evaluation of their efficiency, in *Compatible Materials for the Protection of European Cultural Heritage,* ed. G. Biscontin, A. I. Moropoulou, M. Erdick, and J. Delgado Rodrigues (Athens: Technical Chamber of Commerce, 1998), PACT 56, 149–63.

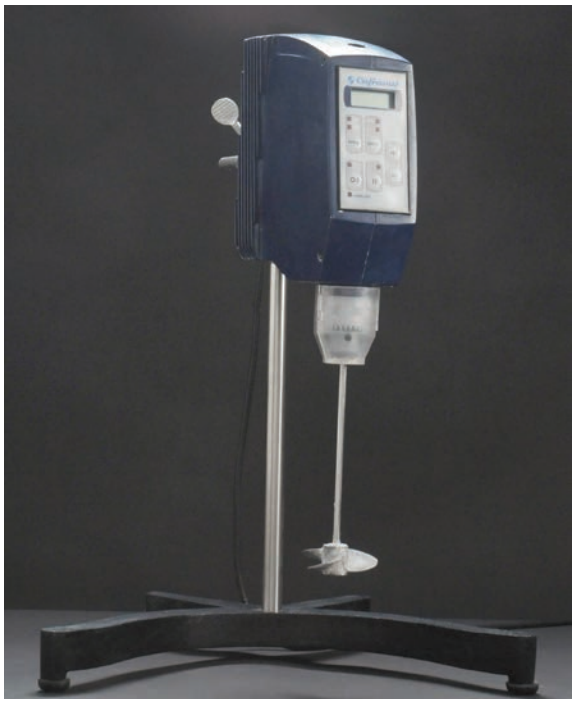


**Figure 1.5.1** A universal variable-speed stirrer with three-blade propeller (Caframo Stirrer BDC 3030) for mixing injection grouts.

PART I

# Laboratory Testing Procedures

# 2. Qualifying Laboratory Testing Procedures



## 2.1. Injectability

### Aim

This test determines the ability of grouts to fill a capillary network of different granular materials, either dry or prewetted, under constant pressure. Injectability is a critical property for injection grouts, which must be suitable for injection through a syringe or tubing to fill internal cracks and voids.

### Description

This test is an adaptation of the sand column test (EN 1771—Determination of Injectability Using the Sand Column Test), to be used for injection grouts for architectural surfaces. The test requires the injectability apparatus (commercially available) shown in figures 2.1.1 and 2.1.2. Grout is injected under constant pressure into a column (fig. 2.1.3), which consists of a transparent plastic tube filled with granular material. The column is mounted vertically, and grout is injected from the bottom of the tube. The distance the grout penetrates into the column and the corresponding time are used to plot a curve that provides a measure of injectability. This test can be used for testing nonhydraulic injection grouts as well.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

Silica sand (EN 196-1: Methods of Testing Cement—Part 1: Determination of Strength) specified in the standard test protocol is replaced by crushed granular materials that are more nearly representative of materials used in archaeological and historic architectural systems. Crushed brick and crushed travertine are used at the GCI. The main reason for this modification is that the absorption capacity

**Figure 2.1.1** Example of a commercially available injectability apparatus.

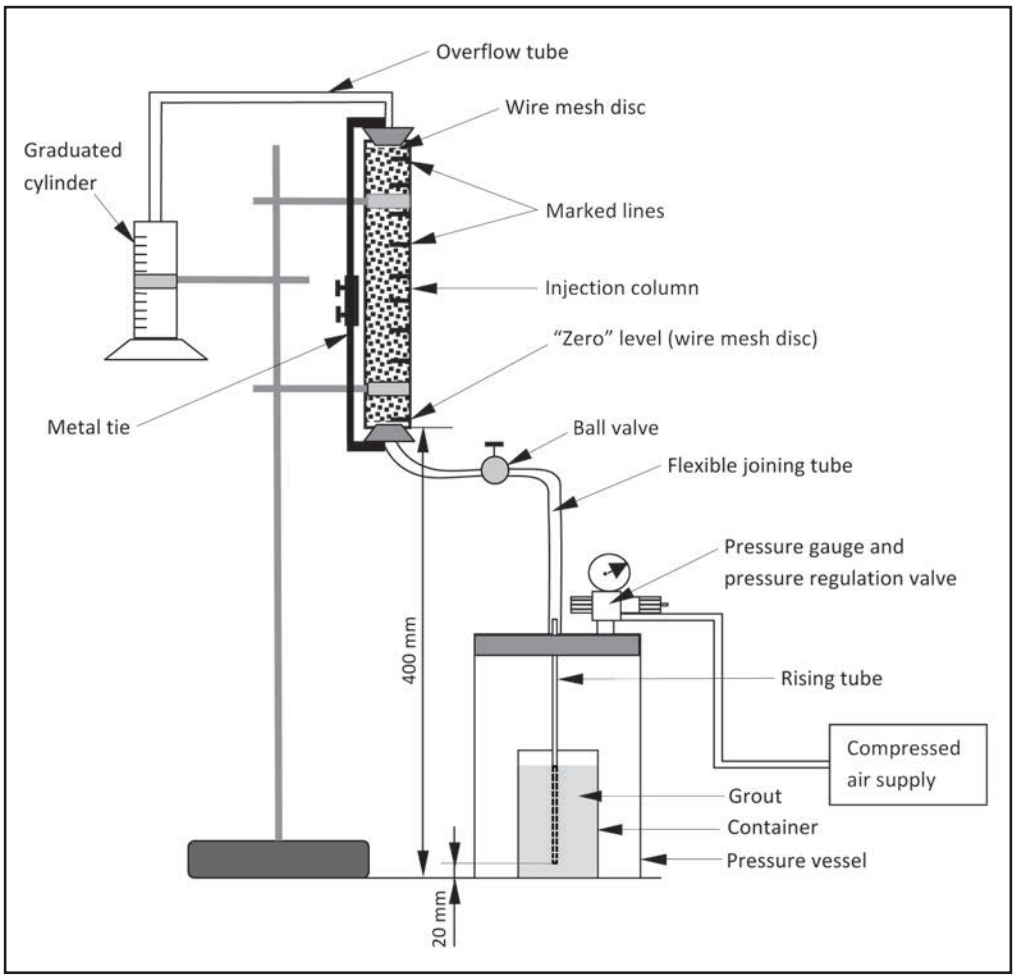



**Figure 2.1.2** Simplified drawing of injectability apparatus (details can be found in EN 1771).

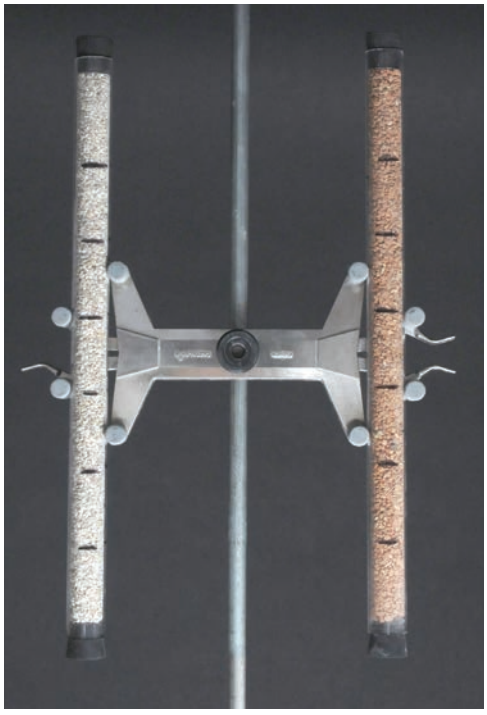


**Figure 2.1.3** Columns filled with crushed brick and crushed travertine.

of EN 196-1 silica sand is low (3% by weight) and does not represent most wall conditions. Crushed brick and crushed travertine are typical of architectural substrates for plasters, wall paintings, and mosaics, and they represent high and low water absorptive media, respectively. The amount of water absorbed after a 10 minute soaking time is approximately 30% by weight for crushed brick and 12% by weight for crushed travertine. It is suggested that testing be carried out with both media when the absorption conditions of the area where the grout will be used are not known.

In addition, the sand suggested in the standard for achieving a flow into a 0.2 mm crack in concrete is replaced by crushed brick and crushed travertine with grain sizes of 2–4 mm, a size that simulates an approximately 0.3–0.6 mm crack width. The particle size distributions of the crushed brick and crushed travertine, as determined by sieve analysis, and the comparison of the particle size distribution of the granular material used in this test method with that of EN 196-1 silica sand are given in figure 2.1.4.

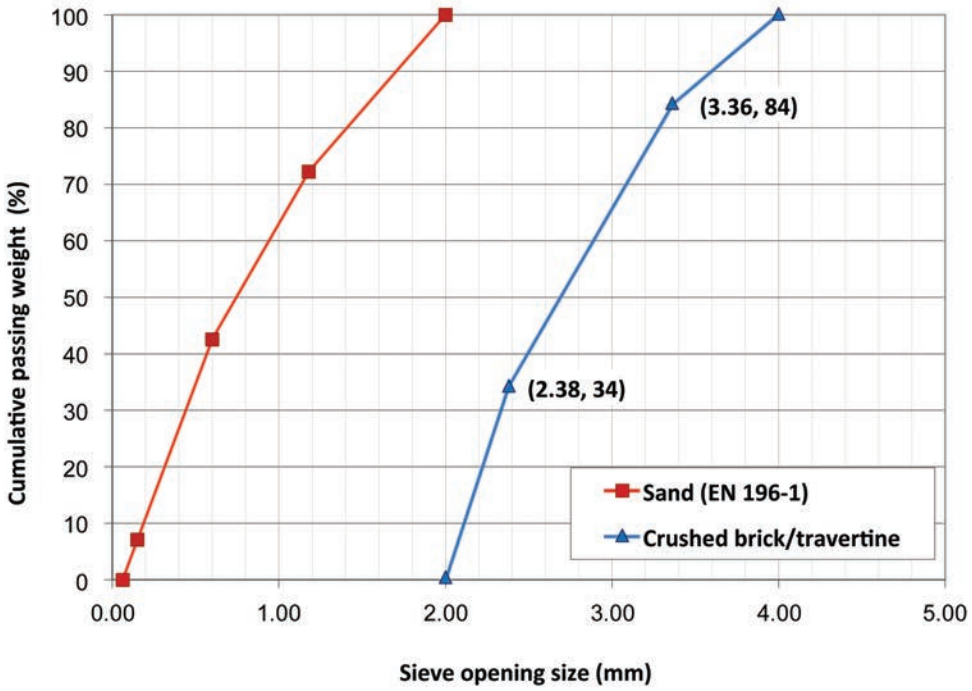



**Figure 2.1.4** Particle size distribution curves of sand (EN 196-1), and crushed brick and crushed travertine.

### Notes

All the materials, test apparatus, and columns are stored under the same conditions (23°C ± 2°C and 60% ± 10% RH) at which the test will be run. The temperature of the grout after mixing and at the end of injection should be kept at 23°C ± 2°C to prevent any temperature change induced variation in the rate of hydration of the grout.

### Equipment and Materials

- injectability apparatus from EN 1771 (figs. 2.1.1 and 2.1.2)
- laboratory stand
- metal tie (fig. 2.1.5) or fabric strap to hold the two perforated stoppers attached to the column in place during testing (the metal tie used at the GCI consists of two aluminum rods, two U-shaped aluminum plates screwed to one end of each rod, and a short aluminum tube connecting the rods; the aluminum tube has two drilled holes to insert two small butterfly screws to secure the position of the rods)
- transparent rigid plastic (e.g., polymethyl methacrylate, PMMA) tube with 22.00 ± 0.05 mm inner diameter and approximately 1.75 mm wall thickness; the length of the tube used is 1.82 m
- 2–4 mm grain size crushed brick (141 ± 2 g per column) and/or crushed travertine (205 ± 2 g per column); see fig. 2.1.4 for gradation details
- 200 mL deionized water, or water and alcohol, per column, if prewetting will be applied
- two 22 mm diameter stainless steel wire mesh discs with 0.5 mm mesh opening per column
- two solid rubber stoppers (size 4) per column
- metal or plastic cylindrical container approximately 80 mm in diameter and 175 mm high
- 25 mL graduated glass cylinder
- 400 mm ruler in millimeters
- stopwatch
- balance accurate to within 0.1 g
- permanent marker

**Figure 2.1.5** Example of a metal tie for the injectability apparatus.

- thermometer accurate to within 1°C
- relative humidity (RH) meter, accurate to within 1%
- tile saw with a PMMA cutting blade
- dessicator and dessicant (e.g., silica gel)
- oven

### Column Preparation

1. Dry the granular materials in an oven at 105°C for 20 hours, and cool the materials to ambient temperature in a desiccator for 4 hours before weighing. Repeat the drying and weighing procedure until a constant weight is reached, which is achieved when the difference between two successive measurements at a 24 hour interval is less than or equal to 0.1%.
2. Cut transparent plastic tube (22.00 ± 0.05 mm inner diameter) into columns 390 mm in length using a tile saw. At least two columns per grout are needed for each medium and injection condition.
3. Weigh the column, including two wire mesh discs and solid rubber plugs to the nearest 0.1 g ($M_1$). Skip this step when the weight of granular material needed to prepare a column has already been determined.
4. Place a wire mesh disc and a rubber plug in the bottom end of the column (fig. 2.1.2).
5. Mark columns at 50 mm intervals using a permanent marker, starting at the bottom wire mesh level, up to a height of 300 mm. Mark the position of the wire mesh disc as "zero" level (fig. 2.1.2).
6. If known, weigh the granular material ($M_f$) needed for each column: 141 ± 2 g for a crushed brick column and 205 ± 2 g for a crushed travertine column.
7. Fill the column with crushed brick or crushed travertine in three equal layers of 120 ± 5 mm in height, to compact granular material evenly (fig. 2.1.3).
8. After filling the first layer, apply 50 lateral shocks by slowly hitting the vertically held column against the edge of a table while holding it from the bottom end. Shocks should be evenly distributed over the height of the layer (10 times, from the bottom to the top of the layer) and the column should be rotated (5 rotations).
9. Repeat step 8 after filling both the second and the third layer.
10. Check the total height of the column. The total height of the compacted crushed brick or crushed travertine in the column should be 360 ± 1 mm. If not, empty the column and repeat the filling procedure. The compaction of the granular material is very important in order to obtain similar capillary networks.
11. Place the wire mesh disc and stopper into the top end of the column (fig. 2.1.2).
12. Weigh the filled column and rubber stoppers to the nearest 0.1 g ($M_2$).
13. Store columns upright vertically until testing is performed.

### Procedure

1. Place the prepared column on the stand. First attach the bottom rubber stopper connected to the flexible joining tube and then the top one connected to the overflow tube (fig. 2.1.2).
2. Secure the columns and the stoppers with the metal or fabric tie (figs. 2.1.2 and 2.1.5).
3. Prepare the grout and pour 500 g of grout into the test container.
4. Measure and record the initial temperature of the grout.
5. Place the container in the pressure vessel (fig. 2.1.2) and close the lid of the vessel.
6. Turn on the compressed air supply with the ball valve on the flexible tube closed; adjust the pressure in the vessel to 0.075 MPa (fig. 2.1.2).
7. Check the vessel for leaks: If the pressure in the vessel decreases, release the pressure, open the vessel lid, reclose it, and repeat step 6.
8. Open the ball valve on the flexible tube (fig. 2.1.2). The experiment should begin within 4 minutes after the completion of mixing.
9. Start the stopwatch when the grout reaches the bottom wire mesh ("zero" level) (fig. 2.1.2).
10. Note the time elapsed when the grout reaches the heights of 50 mm, 100 mm, 150 mm, 200 mm, 250 mm, 300 mm, 360 mm in the column. Continue injection until the column is full and 20 mL excess grout is collected at 1 mL/min in the graduated cylinder, or until the grout rise in the column is slower than 30 mm/min (fig. 2.1.6).
11. When one of the conditions given in step 10 is fulfilled, stop the stopwatch, close the valve on the flexible tube, release the pressure in the vessel, and remove the container including the remaining grout.
12. Measure and record the temperature of the grout. The temperature should be 23°C ± 2°C.
13. Remove the column from the stand: First place the top solid rubber stopper, then turn the column upside down and place the second solid rubber stopper in the bottom end.
14. Weigh the column to 0.1 g ($M_3$).

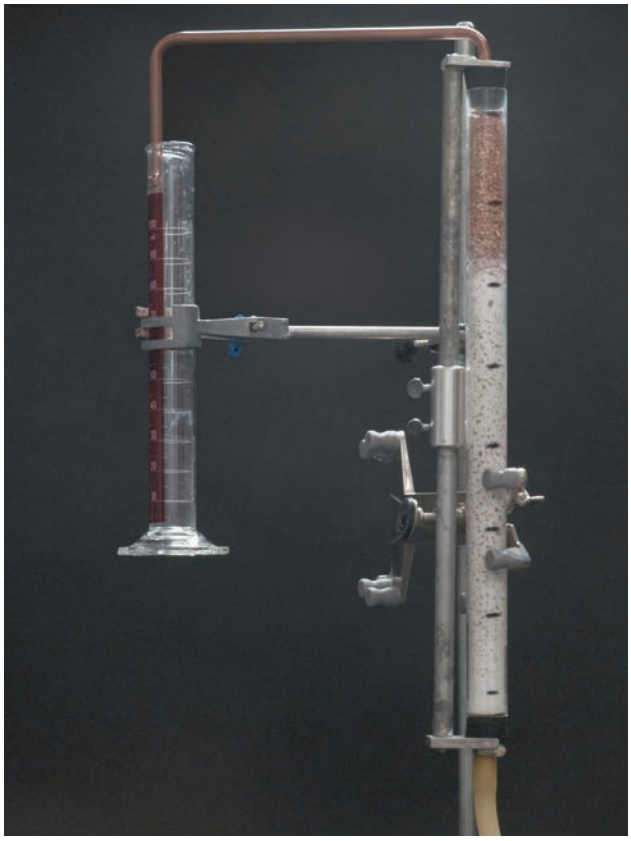


**Figure 2.1.6** Injected grout flowing into the crushed-brick-filled column.

### Prewetting

Prewetting the absorbent granular material before injecting grouts is a common practice. Prewetting can be useful to avoid rapid drying and to ensure proper adhesion. The following steps explain how to prewet the columns to evaluate the effects of prewetting on injectability:

1. Place the prepared column on the stand. First, attach the bottom rubber stopper connected to the flexible joining tube and then the top one connected to the overflow tube (fig. 2.1.2).
2. Secure the column and the stoppers using the metal tie or fabric strap (fig. 2.1.2).
3. Pour 200 mL of deionized water or other prewetting liquid into the test container, place the container into the pressure vessel, and close the lid of the vessel.
4. Turn on the compressed air supply with the ball valve on the flexible tube closed; adjust the pressure in the vessel to 0.075 MPa (fig. 2.1.2).
5. Check the vessel for leaks: If the pressure in the vessel decreases, release the pressure, open the vessel lid, reclose it, and repeat step 4.
6. Open the valve on the flexible tube (fig. 2.1.2).
7. Inject the liquid into the column until 20 mL of it is collected in the graduated cylinder. Stop the injection by closing the valve on the flexible tube.
8. Release the pressure and allow the excess liquid to drain for 10 minutes.
9. Remove and weigh the column (including wire mesh discs and rubber stoppers) ($M_4$).
10. Dry the test container and follow the procedural steps for injecting grout.

### Results

1. Calculate the weight of dry crushed brick or crushed travertine ($M_f$) in the column in grams,

$$M_f = M_2 - M_1 \qquad (2.1.1)$$

where $M_1$ is the weight of the empty column with two wire mesh discs and stoppers, and $M_2$ is the weight of the column filled with crushed brick or crushed travertine and two stoppers, both in grams.

The weight of crushed brick or crushed travertine used to fill the column is an important parameter that indicates the degree of compaction of fill material in each column. It is necessary to determine the weight of granular material needed to prepare a 360 ± 1 mm column for each medium type, and the same amount of filler should be used for the preparation of each column. These values are determined as 141 ± 2 g for the crushed brick and as 205 ± 2 g for the crushed travertine used at the GCI. When other granular material is used during column preparation, the amount of filler in the column should be calculated using equation 2.1.1.

2. Calculate the weight of injected grout ($M_g$),

$$M_g = M_3 - M_2 \qquad (2.1.2)$$

When the granular material in the column is prewetted, the weight of injected grout ($M_g$) is calculated by

$$M_g = M_3 - M_4 \qquad (2.1.3)$$

where $M_2$ is the weight of the column filled with crushed brick or crushed travertine and two stoppers; $M_3$ is the weight of the column after injection, including rubber stoppers; and $M_4$ is the weight of the prewetted column and stoppers, all in grams. The weight of injected grout may be used to compare the injectability of grouts.

3. Draw injectability curves for each grout by plotting the height reached in the column by the grout along

the y-axis and the corresponding measured time along the x-axis:

$$h = func(t) \qquad (2.1.4)$$

where $h$ is the height of the grout in the column in millimeters, and $t$ is the time in seconds taken by the grout to reach the differential reference marks drawn along the column.

Injectability of the grout can also be classified as:

a. Easy (E)—if the column is filled and 20 mL excess grout is collected at 1 mL/min in the graduated cylinder;
b. Feasible (F)—if the column is filled but the 1 mL/min overflow is not achieved;
c. Difficult (D)—if the injected grout is halted or the grout rises more slowly than 30 mm/min before the column is filled. When the grout is classified as "difficult," record the height reached in the column (h) in millimeters and the corresponding time (t) in seconds ($D_{h\text{-}t}$). As an example, injectability curves for three different grouts, all injected into dry-crushed travertine columns, are plotted in figure 2.1.7, and the appropriate category for each grout is indicated on the graph.
4. Repeat the test using individually mixed grouts for each condition until two experimental runs providing the same classification are obtained. The average height and time of the two runs are used as the final values for the grouts classified as difficult. The average height reached in the column for two runs should not differ by more than 25%; otherwise, the test should be repeated.
5. An example data collection sheet is given in figure 2.1.8.

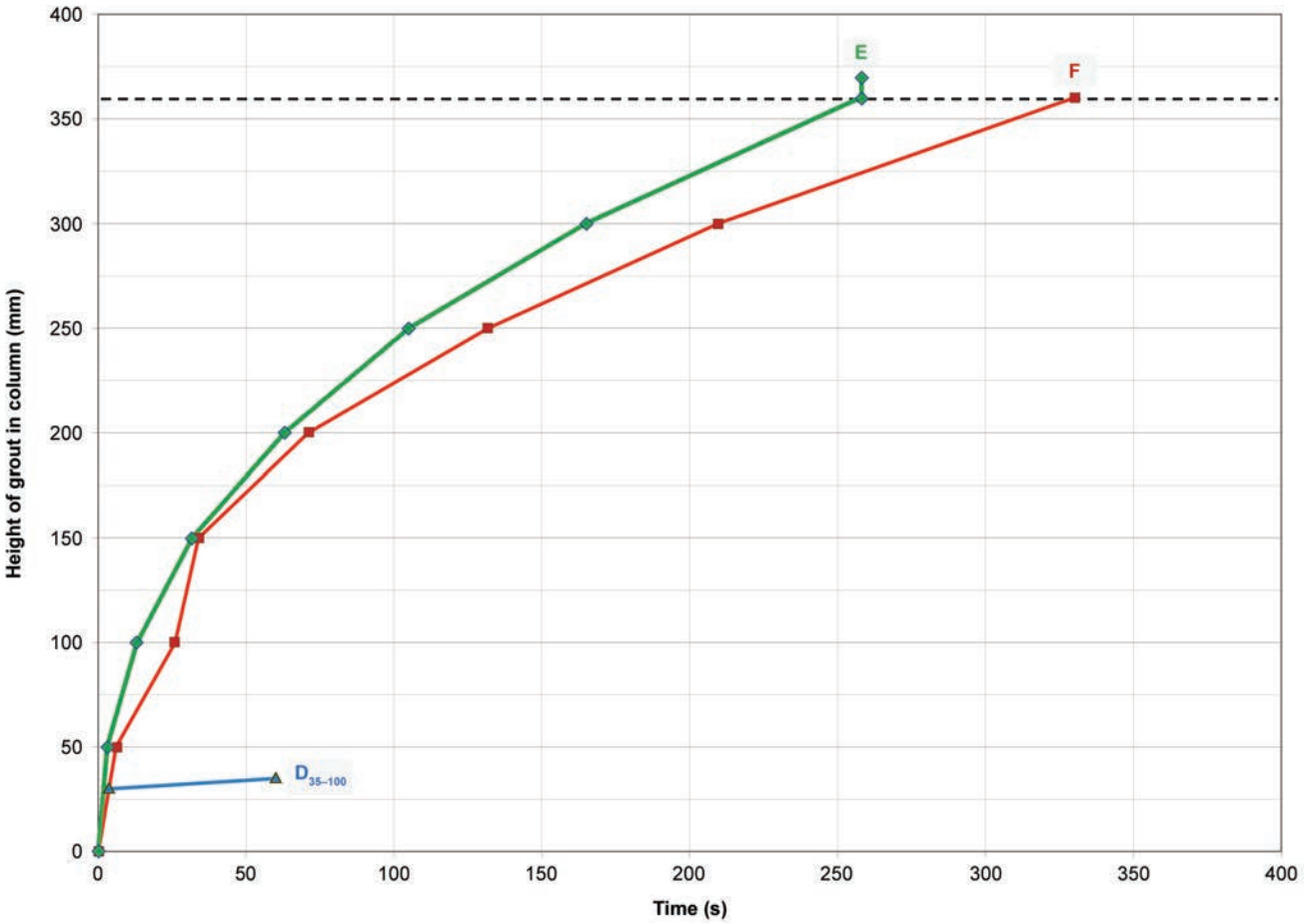



**Figure 2.1.7** Injectability curves for three grouts in dry-crushed travertine columns. The total height of the column filled with the granular medium is 360 mm—indicated as a dashed horizontal line on the plot. Overflow, in case of easy (E) injectability, is indicated by an extra data point at 370 mm.

## INJECTABILITY

| | | | | |
|---|---|---|---|---|
| **Grout name** | | **Grout Temperature (°C)** | **After mixing** | |
| **Grout proportions** | | | **After injection** | |
| **Operator** | | **Date** | | |

**$M_1$ (g):** Weight of the empty column with two wire mesh discs and stoppers
**$M_2$ (g):** Weight of the column filled with crushed brick/travertine, two wire mesh discs, and stoppers
**$M_3$ (g):** Weight of column after injection with two wire mesh discs and stoppers
**$M_4$ (g):** Weight of the prewetted column before injection with two wire mesh discs and stoppers
**$M_f$ (g):** Weight of the crushed brick/travertine in column, calculated using [$M_2$-$M_1$]
**$M_g$ (g):** Weight of the grout in the column, calculated using [$M_3$-$M_2$] or [$M_3$-$M_4$] when prewetted

| | Travertine-wet | | Travertine-dry | | Brick-wet | | Brick-dry | |
|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| $M_1$ (g) | | | | | | | | |
| $M_2$ (g) | | | | | | | | |
| $M_3$ (g) | | | | | | | | |
| $M_4$ (g) | | | | | | | | |
| $M_f$ (g) | | | | | | | | |
| $M_g$ (g) | | | | | | | | |

**h (mm):** Height of the grout in column
**t (s):** Time taken by the grout to reach height h

| | Travertine-wet | | Travertine-dry | | Brick-wet | | Brick-dry | |
|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| h (mm) | t (s) | t (s) | t (s) | t (s) | t (s) | t (s) | t (s) | t (s) |
| 50 | | | | | | | | |
| 100 | | | | | | | | |
| 150 | | | | | | | | |
| 200 | | | | | | | | |
| 250 | | | | | | | | |
| 300 | | | | | | | | |
| 360 | | | | | | | | |

**EASY (E):** If the column is filled and 20 mL excess grout is collected at 1 mL/min in the graduated cylinder
**FEASIBLE (F):** If the column is filled but the 1 mL/min overflow is not achieved
**DIFFICULT (D):** If the injected grout is halted or slower than 30 mm/min before the column is filled; record the final height and time ($D_{h\text{-}t}$)

| | Travertine-wet | | Travertine-dry | | Brick-wet | | Brick-dry | |
|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| Classification | | | | | | | | |

**Figure 2.1.8** Data collection sheet for the injectability test method.

## 2.2. Expansion and Bleeding

### Aim

This test is used to determine the amount of expansion and accumulation of bleed water at the surface of freshly mixed grout. Grouts that are well formulated and properly proportioned should not visibly segregate or bleed. Excessive segregation or bleeding of a grout will change its properties and cause clogging during injection. A suggested amount of final bleeding is less than 0.4%.

### Description

This test is in compliance with ASTM C 940—Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory. Grout is placed in a graduated glass cylinder, and the change in total volume and the accumulation rate of bleed water on the surface of the grout is observed over a period of time. The volume of bleed water as a percentage of the total volume of grout is an indication of the extent of the separation of the liquid and solid phases. It can be used to assess the amount of mixing water for a specific grout. Injection grouts including cement binder generally show expansion due to the heat of hydration (i.e., the expansion of air in the grout released from aggregates, entrapped/entrained during mixing, and/or present in mix water), the formation of hydration products (e.g., ettringite), and alkali content. In contrast, lime-based hydraulic injection grouts show minimal expansion due to these factors. In certain cases, some expansion may be desirable (e.g., in large voids with fragile plaster layers, which require relatively large amounts of grout without the addition of excessive weight). It is important to determine the expansion of a lime-based hydraulic grout when the injection grout includes additives that facilitate expansion and in some cases generate gas (e.g., fine aluminum powder), in order to avoid causing damage to the architectural surfaces. This test can also be used for testing nonhydraulic injection grouts.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

The standard ASTM C 940 procedure is followed, with the exception of the volume of grout used, which is reduced from 800 ± 10 mL to 400 ± 10 mL, since thoroughly mixing 800 mL grout may be a challenge, and for injection grouts 400 mL is adequate.

### Notes

The ambient temperature of the room should be maintained at 23°C ± 2°C. In addition to this, all the dry materials and mixing water should be stored in the same conditions (23°C ± 2°C) at which the test will be run.

### Equipment and Materials

- 500 mL graduated glass cylinder reading to 10 mL
- 25 mL graduated glass cylinder reading to 1 mL
- 10 mL syringe with cannula (#20 or smaller)
- thermometer accurate to within 1°C
- Parafilm or any other type of plastic wrap that will prevent evaporation of bleed water during testing

### Procedure

1. Place the 500 mL graduated cylinder on a level surface that is free from vibration.
2. Mix the grout and immediately measure and record its temperature. The temperature of the grout should be at 23°C ± 2°C.
3. Introduce the grout into the graduated cylinder until the volume of the sample is 400 ± 10 mL (fig. 2.2.1). Volume measurements should begin within 3 minutes after mixing.
4. Cover the top of the graduated cylinder using Parafilm to prevent the evaporation of bleed water.
5. Record the total initial volume of the sample ($V_0$) and the time at which the reading was taken.
6. Record the volume at the upper surface of the bleed water layer ($V_t$) and at the upper surface of the grout

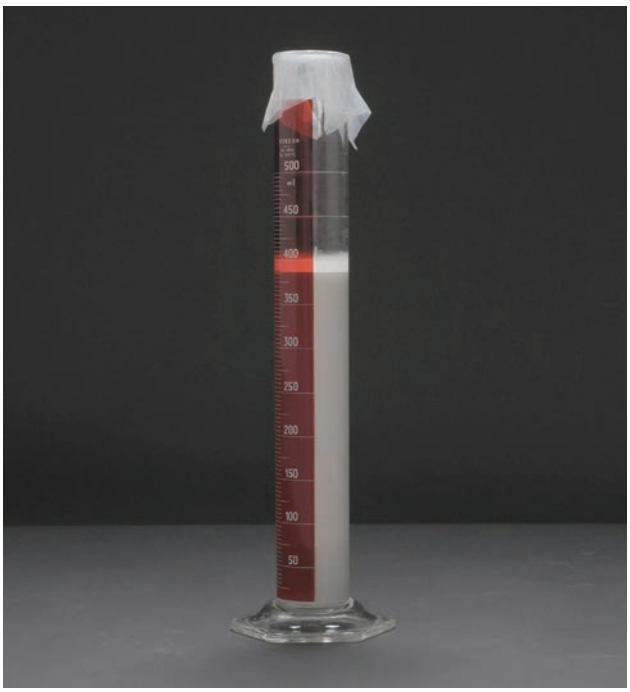



**Figure 2.2.1** Grout in a graduated glass cylinder.

($V_g$), to the nearest 5 mL at 15 minute intervals for the first 60 minutes, and thereafter at hourly intervals until two successive readings show no further change in the volume of the grout.

7. At the end of the test, transfer the bleed water into a 25 mL graduated cylinder by tilting the cylinder and drawing the water off with the syringe. Record the final volume of the bleed water ($V_w$) to the nearest 0.5 mL.

### Results

1. Calculate the expansion of the grout (E) to the nearest 0.2% for each prescribed interval:

$$E = \frac{V_g - V_0}{V_0} \times 100 \qquad (2.2.1)$$

where $V_0$ is the volume of the specimen at the beginning of the test, and $V_g$ is the volume of the grout portion of the specimen at prescribed intervals, measured at the upper surface of the grout, both in milliliters.

2. Calculate the combined expansion of the grout (CE) to the nearest 0.2% for each prescribed interval:

$$CE = \frac{V_t - V_0}{V_0} \times 100 \qquad (2.2.2)$$

where $V_0$ is the volume of the specimen at the beginning of the test, and $V_t$ is the volume of the specimen at prescribed intervals, measured at the upper surface of the water layer, both in milliliters.

3. Calculate the bleeding of the grout (B) to the nearest 0.2% for each prescribed interval:

$$B = \frac{V_t - V_g}{V_0} \times 100 \qquad (2.2.3)$$

where $V_0$ is the volume of the specimen at the beginning of the test; $V_t$ is the volume of the specimen at prescribed intervals, measured at the upper surface of the water layer; and $V_g$ is the volume of the grout portion of the specimen at prescribed intervals, measured at the upper surface of the grout, all in milliliters.

4. Calculate the final bleed water (FB) as a percentage of the initial volume of the grout, to the nearest 0.2%:

$$FB = \frac{V_w}{V_0} \times 100 \qquad (2.2.4)$$

where $V_0$ is the volume of the specimen at the beginning of the test, and $V_w$ is the volume of decanted bleed water, both in milliliters.

5. The average of two independent measurements using two grout batches is stated as E, CE, and B at each interval, and the average of two independent measurements is stated as FB. The average value should not differ more than 1% from each measured value; otherwise, the test should be repeated.
6. An example data collection sheet for this procedure is given in figure 2.2.2.

## EXPANSION AND BLEEDING

| | | | | |
|---|---|---|---|---|
| **Grout name** | | **Temperature (°C)** | **Room** | |
| **Grout proportions** | | | **Grout** | |
| **Operator** | | **Date** | | |

**$V_0$ (mL):** Volume of the sample at the beginning of the test
**$V_t$ (mL):** Volume of the sample at prescribed intervals, measured at the upper surface of water layer
**$V_g$ (mL):** Volume of grout portion of sample at prescribed intervals, measured at the upper surface of grout
**$V_w$ (mL):** Volume of decanted bleed water

### EQUATIONS

| | |
|---|---|
| Expansion, E (%) = $\frac{V_g - V_0}{V_0} \times 100$ | Combined expansion, CE (%) = $\frac{V_t - V_0}{V_0} \times 100$ |
| Bleeding, B (%) = $\frac{V_t - V_g}{V_0} \times 100$ | Final bleeding, FB (%) = $\frac{V_w}{V_0} \times 100$ |

| | Test 1 | | | | | Test 2 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Hour:min** | **$V_t$ (mL)** | **$V_g$ (mL)** | **E (%)** | **CE (%)** | **B (%)** | **$V_t$ (mL)** | **$V_g$ (mL)** | **E (%)** | **CE (%)** | **B (%)** |
| **0:15** | | | | | | | | | | |
| **0:30** | | | | | | | | | | |
| **0:45** | | | | | | | | | | |
| **1:00** | | | | | | | | | | |
| **2:00** | | | | | | | | | | |
| **3:00** | | | | | | | | | | |
| **4:00** | | | | | | | | | | |
| **5:00** | | | | | | | | | | |
| **6:00** | | | | | | | | | | |
| **7:00** | | | | | | | | | | |
| **8:00** | | | | | | | | | | |

| | Test 1 | Test 2 |
|---|---|---|
| **$V_0$ (mL)** | | |
| **$V_w$ (mL)** | | |
| **FB (%)** | | |

**Figure 2.2.2** Data collection sheet for determining the expansion and bleeding of freshly mixed grouts.

## 2.3. Wet Density

### Aim

This test is used to determine the density of wet grout. Wet density becomes an important parameter if added weight as a result of grouting might cause structural instability or failure of the architectural surfaces. Examples of this would be the grouting of large voids behind architectural surfaces on ceilings and vaults. The wet density test procedure is placed in qualifying laboratory testing methods since it is also needed for drying shrinkage calculations.

### Description

This test follows ASTM C 185—Standard Test Method for Air Content of Hydraulic Cement Mortar (sect. 9.4). A container is filled with grout and weighed. The volume of the container and the measured weight of the grout are used to calculate the wet density. This test can also be used for measuring the wet density of nonhydraulic injection grouts.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

The standard procedure is followed, with the exception of compacting the grout in the cylindrical cup. In the standard, mortar is placed in the cup in three equal layers, and each layer is compacted with a tamper. In this procedure, the grout is poured slowly into the cup until the cup is filled, and compaction is not used since grouts are less viscous than mortars, and the additional compaction resulting from the use of a tamper can lead to air entrapment. When a more viscous grout cannot be transferred to the cup in a continuous stream, compaction, as explained in the standard procedure, is followed.

### Equipment and Materials

- 400 ± 1 mL cylindrical metal (nonreactive with grouts) cup (fig. 2.3.1) with a depth of approximately 88 mm
- tamper made from nonabsorptive, nonabrasive, and nonbrittle material, with a cross section of 13 × 25 mm and a length of 120–150 mm (fig. 2.3.1)
- trowel with a steel blade 100–150 mm in length, with straight edges
- balance accurate to within 0.01 g
- spoon (if needed)
- paper towel(s)

### Procedure

1. Weigh the empty 400 mL cylindrical cup ($M_0$).
2. Slowly fill the cylindrical cup with grout to avoid any air entrapment during pouring.

**Figure 2.3.1** Cylindrical cup and tamper.

3. Tap the sides of the cup lightly with the side of the tamper, once at each of five different locations approximately equally spaced around the outside of the cup.
4. If the grout is not liquid enough to be poured in a continuous stream, use a spoon to gently place the grout into the cup in three equal layers. Tap each layer 20 times around the inner surface of the measure by positioning the broad side of the tamper parallel to the radius and perpendicular to the inner surface of the cup. Each layer is tamped in one complete revolution.
5. Level the grout flush with the top of the cup by drawing a straightedged trowel across, using a sawing action.
6. Clean the edge of the top of the cup, and wipe off all grout and water adhering to the outside of the cup.
7. Weigh the cup with the grout ($M_t$).

### Results

1. Calculate the weight of grout ($M_g$) in grams using

$$M_g = M_t - M_0 \qquad (2.3.1)$$

where $M_t$ is the total weight of the grout and the cup in grams, and $M_0$ is the weight of the container in grams.

2. Calculate the wet density of grout ($\rho_{wet}$) in g·cm$^{-3}$ by

$$\rho_{wet} = \frac{M_g}{400} \qquad (2.3.2)$$

3. The average of two independent measurements is stated as average wet density. The average value should not differ more than 5% from each measured value; otherwise the test should be repeated.
4. An example data collection sheet for this procedure is given in figure 2.3.2.

## WET DENSITY

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |
| **Time** | |

**$M_0$ (g):** Weight of the cup

**$M_t$ (g):** Total weight of the grout and cup

**$M_g$ (g):** Weight of the grout

**$\rho_{wet}$ (g · cm$^{-3}$):** Wet density of the grout

### EQUATIONS

$$M_g = M_t - M_0$$

$$\rho_{wet} = \frac{M_g}{400}$$

| Specimen no. | $M_0$(g) | $M_t$(g) | $M_g$(g) | $\rho_{wet}$ (g · cm$^{-3}$) |
|---|---|---|---|---|
| 1 | | | | |
| 2 | | | | |

**$\rho_{wet}$, average =** _____ **g · cm$^{-3}$**

**Figure 2.3.2** Data collection sheet for wet density measurements.

## 2.4. Drying Shrinkage

### Aim

This method determines the volumetric shrinkage of grouts upon drying. Volumetric stability of an injection grout is an important parameter since shrinkage directly affects adhesion and durability. This test also provides information on the water content in the grout leading to volumetric instability. While increased water content may appear to improve some properties, such as injectability, it will decrease volumetric stability and lead to the formation of cracks, which in turn will cause loss of the bond between the grout and the substrate layers, and loss of grout strength. Drying shrinkage measured using this method should be less than 4% and preferably should be as low as possible.

### Description

This method follows the procedure for measuring the shrinkage of joint compound as given in ASTM C 474—Standard Test Methods for Joint Treatment Materials for Gypsum Board Construction. A grout specimen of known initial volume is dried. The final volume of the grout specimen is calculated using the weight difference between the weights of the specimen measured in air and submerged in a displacement fluid (e.g., kerosene). Shrinkage is calculated using the initial and final volumes of the specimen. Also, dry density of the grout can be calculated using the final weight measured in air and calculated final volume.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

The only modification made to this method is that the specimen is not dried in an oven as specified in ASTM C 474. Instead, it is dried at 23°C ± 2°C and at 60% ± 5% RH.

### Notes

The displacement fluid is stored in the room where the test will be conducted. The density of the displacement liquid is measured using a liquid pycnometer before testing, and the temperature of the displacement fluid is kept at ±1°C of room temperature during the shrinkage measurements. In the procedure section, the setup used at the GCI for weighing the specimen in air and kerosene is explained in detail. Other setups fulfilling the same function can be prepared, depending on the available equipment in the laboratory.

### Equipment and Materials

- six cylindrical plastic molds (50 mm diameter and at least 20 mm high)
- thin plastic wrap (e.g., food wrap)
- single-pan balance accurate to within 0.01 g (max. 3000 g)
- 1 L beaker, crystallization dish, or wide-mouth glass container
- two U-shaped stands (fig. 2.4.1) with 90° angled corners: one with a rod to hang the wire basket
- stainless steel or noncorrosive metal wire mesh basket (fig. 2.4.2) and thread to hang the basket
- two 20 mL syringes
- kerosene as displacement fluid (CAS no. 8008-20-6)

**Figure 2.4.1** Two U-shaped stands with 90° angled corners. The stand with the rod to hang the basket is on the right.

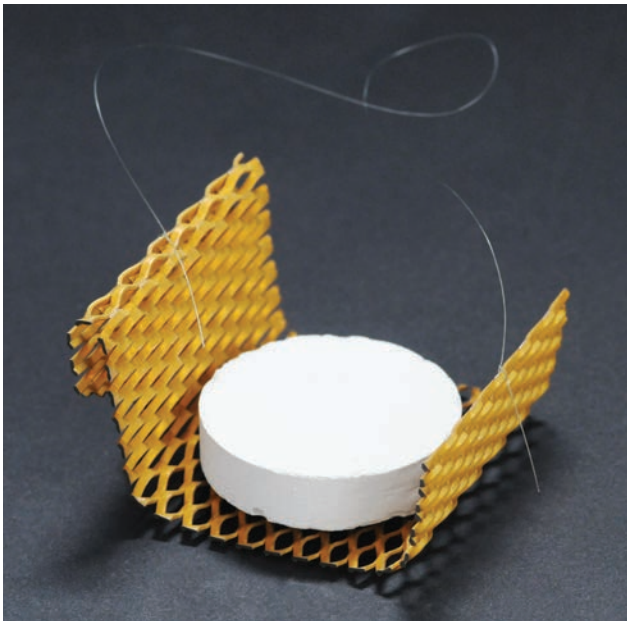


**Figure 2.4.2** Grout specimen and wire basket.

**Figure 2.4.3** Shrinkage specimens molded using plastic sheet.

- weighing boats or papers
- petroleum jelly
- lint-free cloth
- thermometer accurate to within 1ºC
- RH meter, accurate to within 1%
- 25 mL liquid pycnometer

### Specimen Preparation

1. Label the plastic molds and apply small amount of petroleum jelly inside the molds.
2. Place sheets of plastic wrap (approximately 150 mm × 150 mm) into the molds for easy demolding, smooth out the wrinkles using a finger, and weigh the mold and plastic sheet ($M_0$).
3. Fill the syringe with 20 mL grout. Remove entrapped air bubbles by tapping the syringe with a finger.
4. Inject 20 mL grout into the molds and record the total weight (M) (fig. 2.4.3).
5. Allow the specimens to dry at 23°C ± 2°C and 60% ± 5% RH for at least 4 weeks or preferably until the constant weight is reached, which is achieved when the difference between two successive measurements at a 24 hour interval is less than or equal to 0.1% of the weight of the specimen.
6. One week into drying, demold the specimens by pulling the ends of the plastic sheet. Carefully separate the specimens from the plastic sheet, turn them over, and place back on the plastic sheet. Place the labeled empty mold onto the plastic sheet holding the specimen.

### Procedure

1. Place the U-shaped stand with the hanging rod on the pan of the balance, as shown in figures 2.4.4 and 2.4.5.

**Figure 2.4.4** Drying shrinkage test.

2. Position the second U-shaped stand on the balance, spanning the balance pan as shown in figures 2.4.4 and 2.4.5.
3. Measure the temperature and the relative humidity of the room.
4. Measure the temperature of the kerosene.
5. Measure the density of the kerosene using the pycnometer as follows: Weigh a dry pycnometer and its cap with a capillary overflow tube ($M_{pyc}$). Fill the pycnometer with kerosene using the second 20 mL syringe and close the cap. Clean the excess kerosene and reweigh ($M_{pyc+k}$).

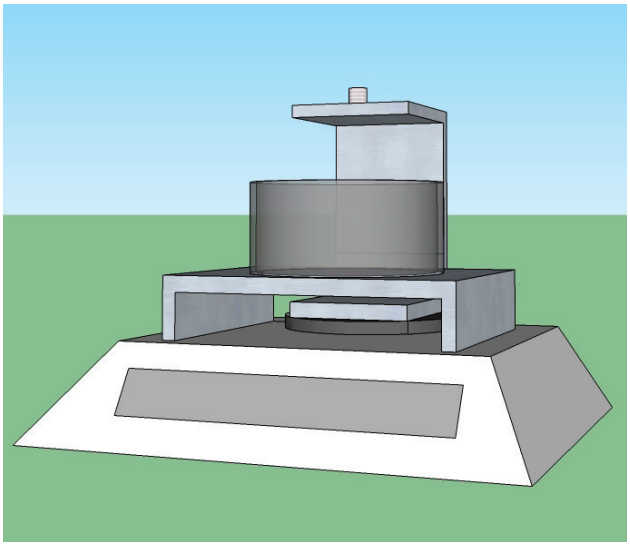


**Figure 2.4.5** Simplified drawing of drying shrinkage test setup.

6. Fill the 1 L beaker or other container with kerosene and place it on the stand as shown in figures 2.4.4 and 2.4.5.
7. Hang the wire basket (fig. 2.4.4) such that it is immersed in kerosene and does not touch the sides of the beaker.
8. Tare the balance.
9. Weigh a grout specimen ($M_{A1}$) to 0.01 g by placing the specimen on top of the U-shaped stand, next to the rod used for hanging the wire basket (fig. 2.4.4). If the grout specimen is broken into pieces because of the demolding and/or drying shrinkage, weigh all the large pieces and leave the rest of the specimen on the plastic sheet.
10. Transfer the grout left on the plastic sheet (scrub the surface of the sheet, if needed) into a labeled weighing boat or paper.
11. Place the grout specimen including all the weighed pieces into the wire basket suspended in kerosene (fig. 2.4.4), such that both are totally immersed in kerosene and the wire basket does not touch the sides of the beaker, until a constant weight is reached.
12. Remove the specimen from the kerosene. Lightly blot excess fluid off the surface of the specimen with a cloth moistened in the same fluid.
13. Place the specimen on top of the U-shaped stand, next to the rod used for hanging the wire basket (fig. 2.4.4) and weigh the specimen in air ($M_{A2}$) to 0.01 g.
14. Place the specimen back into the wire basket (figure 2.4.4). Ensure that they are completely immersed in kerosene and that the wire basket does not touch the sides of the beaker. Record the weight ($M_{im}$) to 0.01 g.
15. Complete the calculations in step 1 in the results section before testing the rest of the specimens if the appropriateness of displacement fluid has not been determined for the tested grout.
16. Repeat procedural steps 8–14 using the rest of the specimens. Check the position of the wire basket so that it is immersed in kerosene and does not touch the sides of the beaker before testing each specimen.
17. Remove the setup from the balance, place an empty weighing boat or paper, and tare the balance.
18. Transfer the grout in labeled weighing boat or paper into empty weighing boat or paper placed on the balance, and record the weight ($M_L$) for each specimen.

**Results**

1. To ensure that the displacement fluid used is appropriate for the grout being tested, the percent weight increase of the grout specimen as a result of absorbing the displacement fluid (ΔM), in percentage (%), is calculated by using

$$\Delta M = \frac{M_{A2} - M_{A1}}{M_{A1}} \quad (2.4.1)$$

where $M_{A1}$ is the weight of the specimen in air before being immersed in kerosene, and $M_{A2}$ is the weight of the specimen in air after being immersed in kerosene, both in grams.

If ΔM is more than 25%, the procedural steps 4–15 should be repeated using a different displacement fluid and specimen, since the absorption of the displacement fluid may lead to the expansion of the specimen and therefore may cause the reduction of the calculated volumetric shrinkage.

2. The weight difference and the volume of the pycnometer ($V_{pyc}$) are used to calculate the density of the kerosene ($\rho_k$) as follows:

$$\rho_k = \frac{M_{pyc+k} - M_{pyc}}{V_{pyc}} \quad (2.4.2)$$

where $M_{pyc}$ is the weight of the dry pycnometer and its cap in grams, $M_{pyc+k}$ is the weight of the kerosene-filled pycnometer and its cap in grams, and $V_{pyc}$ is the volume of the pycnometer in cubic centimeters ($cm^3$).

3. The initial volume of the specimen ($V_i$) in cubic centimeters is calculated using

$$V_i = \left(\frac{M - M_0}{\rho_{wet}}\right)\left(\frac{M_{A1}}{M_{A1} + M_L}\right) \quad (2.4.3)$$

where $M$ is the weight of the fresh grout, plastic mold, and plastic sheet, $M_0$ is the weight of the mold and plastic sheet, both in grams, $\rho_{wet}$ is the wet density of the grout in grams per cubic centimeter ($g \cdot cm^{-3}$), $M_{A1}$ is the weight of the specimen in air before being immersed in kerosene, and $M_L$ is the total weight of the small pieces of the specimen and the grout stuck on the plastic sheet, both in grams.

Determination of $\rho_{wet}$ is given in laboratory procedure 2.3.

4. The weight difference ($M_{diff}$) between the specimen in kerosene ($M_{im}$) and in air ($M_{A2}$) is calculated using

$$M_{diff} = M_{A2} - M_{im} \quad (2.4.4)$$

where $M_{A2}$ is the weight of the specimen in air after being immersed in kerosene and $M_{im}$ is the weight of the specimen in kerosene, all in grams.

5. The final volume of the specimen after drying ($V_f$), in cubic centimeters, is calculated using

$$V_f = \frac{M_{diff}}{\rho_k} \tag{2.4.5}$$

where $M_{diff}$ is the weight difference of the specimen, in kerosene and in air, in grams, and $\rho_k$ is the density of kerosene measured using a pycnometer, in grams per cubic centimeter.

6. The percent shrinkage (S) is calculated as

$$S = \frac{V_i - V_f}{V_i} \times 100 \tag{2.4.6}$$

where $V_i$ is the volume of the specimen when the grout is still fresh, and $V_f$ is the volume of the same grout specimen after drying, both in cubic centimeters.

7. The dry density of the grout ($\rho_{dry}$) is calculated as

$$\rho_{dry} = \frac{M_{A1}}{V_f} \tag{2.4.7}$$

where $M_{A1}$ is the weight of the specimen in air before being immersed in kerosene, in grams, and $V_f$ is the volume of the same grout specimen after drying, in cubic centimeters.

8. Calculate the average drying shrinkage and dry density using the results of all the tested specimens. Discard any individual result if the average and the independent measurement differ more than 5%. The average drying shrinkage and dry density should be obtained using the results of at least three specimens; otherwise, the test should be repeated.
9. An example data collection sheet is given in figure 2.4.6.

## DRYING SHRINKAGE

| Grout name | | Room | Temperature (°C) | |
|---|---|---|---|---|
| Grout proportions | | | RH (%) | |
| Operator | | $\rho_{wet}$ | | |

**$\rho_{wet}$ ($g \cdot cm^{-3}$):** Wet density of grout (from laboratory testing procedure 2.3)
**$T_k$ (°C):** Temperature of the kerosene
**$M_{pyc}$ (g):** Weight of empty pycnometer with cap
**$M_{pyc+k}$ (g):** Weight of pycnometer filled with kerosene (with cap)
**$V_{pyc}$ ($cm^3$):** Volume of empty pycnometer with cap
**$\rho_k$ ($g \cdot cm^{-3}$):** Density of kerosene
**$M_0$ (g):** Weight of plastic mold and sheet
**M (g):** Weight of fresh grout sample, plastic mold, and plastic sheet
**$M_{A1}$ (g):** Weight of hardened grout specimen in air before immersed in kerosene
**$M_{A2}$ (g):** Weight of hardened grout specimen in air after immersed in kerosene
**$M_L$ (g):** Total weight of the small pieces of the specimen and grout stuck on the plastic sheet
**$M_{im}$ (g):** Weight of hardened grout specimen in kerosene
**$\Delta M$ (%):** Percent weight increase of grout specimen due to absorption of kerosene
**$M_{diff}$ (g):** Weight difference of the specimen weighed in kerosene and in air
**$V_i$ ($cm^3$):** Volume of specimen before drying
**$V_f$ ($cm^3$):** Volume of specimen after drying
**$\rho_{dry}$ ($g \cdot cm^{-3}$):** Dry density of grout
**S (%):** Volume shrinkage

## EQUATIONS

| | | | |
|---|---|---|---|
| $\rho_k = \frac{M_{pyc+k} - M_{pyc}}{V_{pyc}}$ | $\Delta M = \frac{M_{A2} - M_{A1}}{M_{A1}}$ | $M_{diff} = M_{A2} - M_{im}$ | $V_f = \frac{M_{diff}}{\rho_k}$ |
| $V_i = \left(\frac{M - M_0}{\rho_{wet}}\right) \times \left(\frac{M_{A1}}{M_{A1} + M_L}\right)$ | | $S = \frac{V_i - V_f}{V_i} \times 100$ | $\rho_{dry} = \frac{M_{A1}}{V_f}$ |

| $T_k$ (°C) | $M_{pyc}$ (g) | $M_{pyc+k}$ (g) | $V_{pyc}$ ($cm^3$) | $\rho_k$ ($g \cdot cm^{-3}$) |
|---|---|---|---|---|
| | | | | |

| Date | | | | | | | |
|---|---|---|---|---|---|---|---|
| Specimen no. | $M_0$ (g) | M (g) | $M_{A1}$ (g) | $M_{A2}$ (g) | $M_L$ (g) | $M_{im}$ (g) | $\Delta M$ (%) |
| 1 | | | | | | | |
| 2 | | | | | | | |
| 3 | | | | | | | |
| 4 | | | | | | | |
| 5 | | | | | | | |
| 6 | | | | | | | |

| Specimen no. | $M_{diff}$ (g) | $V_i$ ($cm^3$) | $V_f$ ($cm^3$) | $\rho_{dry}$ ($g \cdot cm^{-3}$) | S (%) |
|---|---|---|---|---|---|
| 1 | | | | | |
| 2 | | | | | |
| 3 | | | | | |
| 4 | | | | | |
| 5 | | | | | |
| 6 | | | | | |

$\rho_{dry}$, average = _____ $g \cdot cm^{-3}$ S, average = _____ %

**Figure 2.4.6** Data collection sheet for the drying shrinkage test method.

## 2.5. Splitting Tensile Strength

### Aim

This test is used to determine the tensile strength of hardened grouts by means of an indirect tension test called a splitting tensile test or a Brazilian test. Grouts used for reattachment of architectural surfaces are expected to fail most of the time due to tensile stresses and sometimes due to shear stresses. Therefore, even though testing for compressive strength is the most common mechanical strength test, in the case of grouts used for architectural surfaces, their tensile and shear strengths are more important for their evaluation than their compressive strength. In this manual, testing for splitting tensile strength is preferred over testing for shear strength since it is the dominant parameter for most applications. Additionally, the splitting tensile strength test uses the same test specimen and testing equipment as the compression strength test for which most laboratories are equipped. The tensile strength of the cured injection grout should be similar to, but less than, the original plaster and substrate to avoid cracking of the original material. It should be noted that the tensile strength obtained from the splitting test and flexural test (another indirect method) is assumed to be around 10% and 50%, higher respectively, than direct tensile strength values. This test does not provide information regarding bond strength.

### Description

The test to determine splitting tensile strength is carried out on a cylindrical specimen that is tested on its side in diametric compression loading. Since the grouts are much weaker in tension than compression, the cylinder will typically fail in response to the horizontal tension forces, rather than in response to the vertical compression forces. This failure leads to a splitting of the cylinder down the middle. Cylindrical specimens are prepared and tested following EN 1771—Determination of Injectability Using the Sand Column Test. An apparatus (figs. 2.5.1 and 2.5.2) is used to fill an empty transparent plastic column with grout under constant pressure. The column is held vertically, and grout is injected from the bottom of the tube. The grout specimens are removed from the column with a tile saw after curing is complete.

**Figure 2.5.1** Example of a commercially available injectability apparatus with an empty column.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

The casting of individual cylindrical specimens of injection grouts, even using the smallest standard cylindrical mortar specimen molds, 50.8 ± 0.25 mm (2.00 ± 0.01 in.) in diameter and 101.60 ± 0.25 mm (4.00 ± 0.01 in.) in height, is problematic due to the higher drying shrinkage of injection grouts compared to mortars and concrete. Current mortar and concrete standard tests (ASTM C 496—Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens) use a large enough sample volume to minimize any effect of size, because when the specimen size of a brittle, heterogeneous material decreases, the probability of having defects also decreases and the strength increases. In addition to this, the use of cylinders having a $d/d_{max}$ (diameter of a cylindrical specimen/maximum aggregate size) ratio larger than 3 minimizes wall effects (ASTM C192-07—Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory). Considering that the maximum aggregate size of injection grouts for architectural surfaces is less than 1 mm and smaller specimens will have less internal cracking due to shrinkage, the size of the cylindrical specimens used in this test is reduced to 22.00 ± 0.05 mm in diameter and 44.00 ± 1.0 mm in height. The challenge of placing and compacting the grout into smaller-diameter individual molds is resolved by injecting the grout into a plastic tube long enough to yield 6 specimens that can be obtained by dry-cutting with a tile saw following EN 1771. For this test, grout is injected into empty columns, since the strength of granular material filling the column and its absorption capacity directly affect the splitting tensile strength.

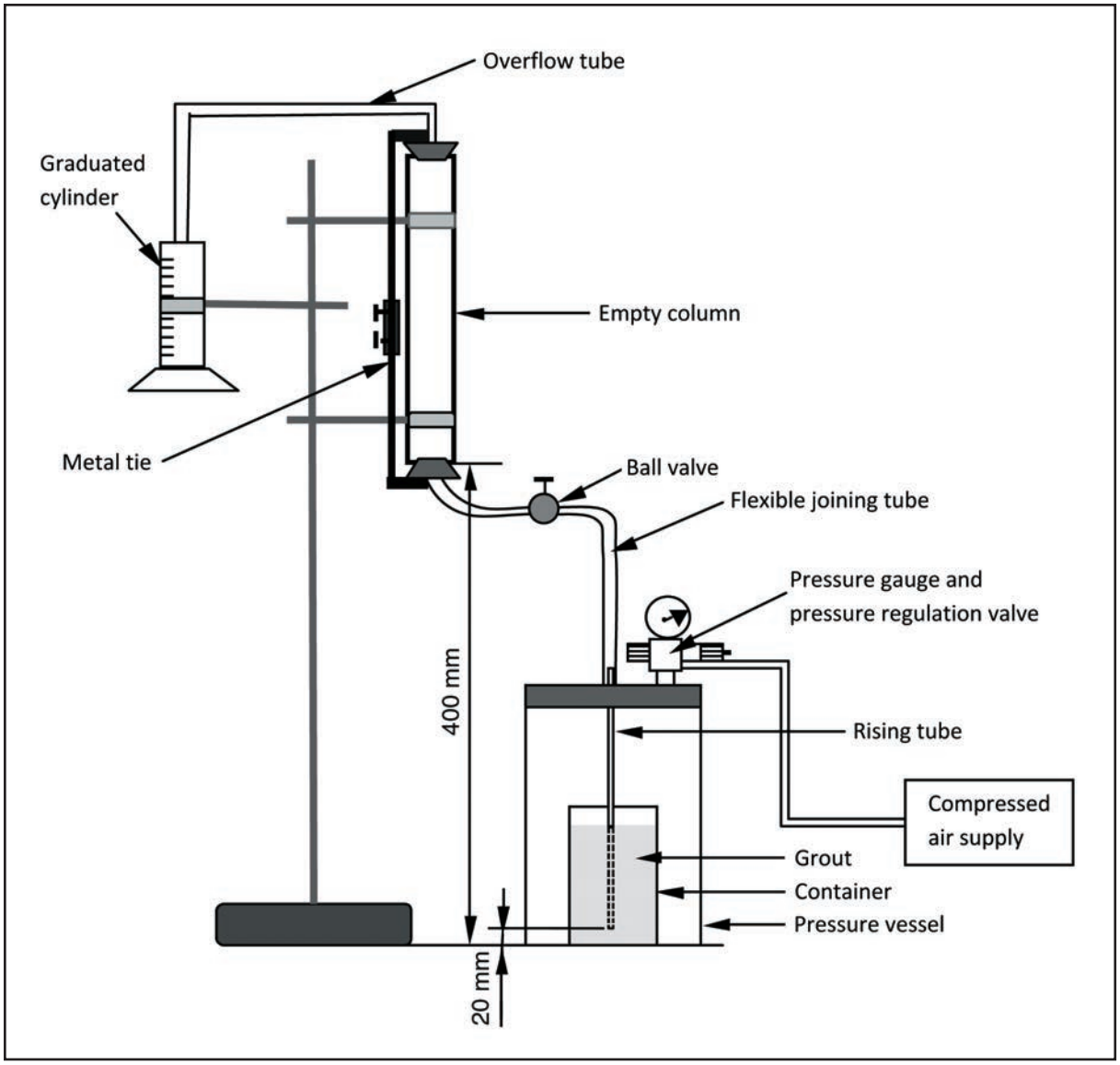



**Figure 2.5.2** Simplified drawing of injectability apparatus (details can be found in EN 1771).

### Notes

All materials, the test apparatus, and columns are stored under the same conditions (23°C ± 2°C and 60% ± 10% RH) at which the test will be run.

### Equipment and Materials

- injectability apparatus from EN 1771 (figs. 2.5.1 and 2.5.2)
- laboratory stand
- metal tie (fig. 2.5.3) or fabric strap to hold the two perforated stoppers attached to the column in place during specimen preparation (the metal tie used at the GCI consists of two aluminum rods, two U-shaped aluminum plates screwed to one end of

**Figure 2.5.3** Example of a metal tie for the injectability apparatus.

each rod, and a short aluminum tube connecting the rods; the aluminum tube has two drilled holes to insert two small butterfly screws to secure the position of the rods)
- transparent rigid plastic (e.g., polymethyl methacrylate, PMMA) tube with 22.00 ± 0.05 mm inner diameter and approximately 1.75 mm wall thickness; the length of the tube used is 1.82 m
- two solid rubber stoppers (size 4)
- metal or plastic cylindrical container approximately 80 mm in diameter and 175 mm high
- 25 mL graduated glass cylinder
- caliper with a resolution of 0.01 mm
- 4 mil plastic sheet (e.g., window insulation sheets)
- 12 wooden strips (5 mm wide, 40 mm long, 1 mm thick)
- adhesive tape
- Instron universal mechanical testing machine or any mechanical testing equipment capable of compressive loading
- tile saw equipped with a PMMA cutting blade
- V-shaped specimen holder for cutting
- thermometer accurate to within 1°C
- RH meter accurate to within 1%

### Column Preparation

1. Cut transparent plastic tube into 300 mm long columns using a tile saw.

### Specimen Preparation

1. Place the empty column on the stand. First, attach the bottom rubber stopper connected to the flexible joining tube and then attach the top one connected to the overflow tube (fig. 2.5.2).
2. Secure the column and the stoppers using the metal tie or fabric strap (fig. 2.5.2 and fig. 2.5.3).
3. Prepare the grout and pour 500 g of the mix into the test container.
4. Place the container in the pressure vessel (fig. 2.5.2) and close the lid of the vessel.
5. Turn on the compressed air supply with the ball valve on the flexible tube closed; adjust the pressure in the vessel to 0.05 MPa (fig. 2.5.2), a lower pressure than that used in the injectability test, since there is no back pressure created by granular material.
6. Check the vessel for leaks: If the pressure in the vessel decreases, release the pressure, open the vessel lid, reclose it, and repeat step 5.

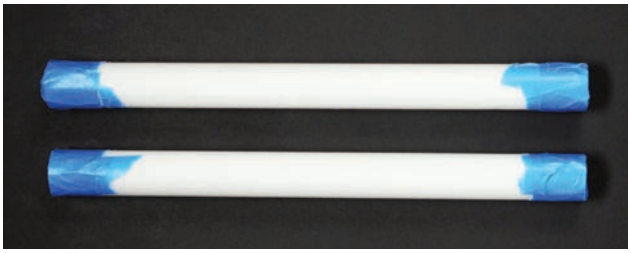
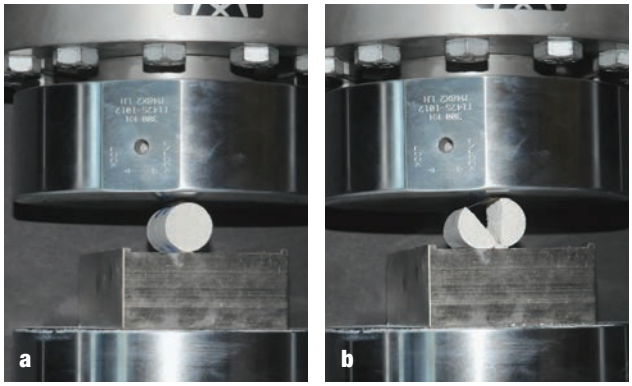


**Figure 2.5.4** Columns filled with grouts.

7. Open the ball valve on the flexible tube (fig. 2.5.2). The experiment should begin within 4 minutes of completion of the mixing.
8. Continue injection until the grout has filled the column.
9. Close the valve on the flexible tube and release the pressure in the vessel.
10. Remove the column from the stand, and seal the column using two solid rubber stoppers and adhesive tape: First, place the top solid rubber stopper and tape it, then slowly turn the column upside down and place the second solid rubber stopper and tape it (fig. 2.5.4).
11. Store the columns vertically, ensuring that the bottoms of the columns during injection remain the bottoms during storage.
12. Cure the columns under sealed conditions at 23°C ± 2°C until the test date. Specimens are tested after at least 2 months or, preferably, after 6 months of curing.
13. Saw the column perpendicular to its axis to obtain 6 cylindrical specimens with an l/d (length to diameter) ratio of 2—i.e., the length is 44.0 ± 1.0 mm.

**Figure 2.5.5** Slitting of a cylindrical plastic tube with the tile saw. The specimen is held and positioned under the saw by means of a V-shaped holder.

**Figure 2.5.6** Wooden strips attached to a specimen.




**Figures 2.5.7a and 2.5.7b** Splitting tensile strength testing (a), and the tested specimen showing the splitting (b).

14. While the specimen is held and positioned using a V-shaped holder, extrude each specimen from the plastic tube by sawing the plastic tube covering the cylindrical specimen along the length without making any saw marks on the specimen (fig. 2.5.5), or simply by pushing the specimen with a finger from one end when possible.
15. Record the position of the specimen in the column.
16. Measure the length (l) of each specimen and diameter (d) to the nearest 0.1 mm using the caliper.

### Procedure

1. Test the specimens within 1 hour of being removed from the plastic tube. Keep the untested specimens covered with a plastic sheet during testing to minimize additional drying.
2. Tape two wooden strips along the top and bottom sides of the sample (fig. 2.5.6) and apply the load through them. Tape should not go all the way around the sides of the specimen.
3. Test the specimens at a constant loading rate of $0.05 \pm 0.01$ N·mm$^{-2}$·s$^{-1}$ in compression (figs. 2.5.7a and 2.5.7b).

### Results

1. Record the maximum load during the test as the breaking load, F, in newtons (N), and calculate the splitting tensile strength, f, in newtons per square millimeter (N·mm$^{-2}$), using the following relationship:

$$f = \frac{2 \times F}{\pi \times d \times \ell} \qquad (2.5.1)$$

   where $d$ is the specimen diameter, and $l$ is the specimen length, both in millimeters.
2. Calculate the average splitting tensile strength using the results of 6 specimens obtained for each grout. Discard any individual result if the average and the individual result differ by more than 20%. The average splitting tensile strength should be obtained for at least 3 individual results; otherwise, the test should be repeated.
3. An example data collection sheet is given in figure 2.5.8.

## SPLITTING TENSILE STRENGTH

| | | | |
|---|---|---|---|
| **Grout name** | | **Compressive loading rate** | 0.05 ± 0.01 $N \cdot mm^{-2} \cdot s^{-1}$ |
| **Grout proportions** | | **Age of column (days)** | |
| **Operator** | | **Date** | |

**F (N):** Breaking load
**d (mm):** Specimen diameter
**l (mm):** Specimen length
**f ($N \cdot mm^{-2}$):** Splitting tensile strength

**EQUATION**

$$f = \frac{2 \times F}{\pi \times d \times l}$$

| Specimen no. | d (mm) | l (mm) | F (N) | f ($N \cdot mm^{-2}$) |
|---|---|---|---|---|
| 1 (top) | | | | |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 (bottom) | | | | |

**f, average =** _____ $N \cdot mm^{-2}$

**Figure 2.5.8** Data collection sheet for the splitting tensile strength test method.

## 2.6. Soluble Salt Content by Ion Chromatography

### Aim

The aim of this test is to determine the soluble salt content of hardened injection grouts. The soluble salt content of the grouts should be as low as possible to avoid damage to original architectural surfaces and elements from leaching salt.

### Description

The method loosely follows the sample preparation suggestions of Christine Bläuer Böhm, in her paper "Assessment of Quantitative Salt Analysis by the Water Extraction Method on Lime Mortars,"[1] and the analysis procedure of ASTM D 4327—Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography. Dried, preconditioned, and finely crushed, hardened grout is added to ultrapure water with a resistivity not greater than 18.2 Mohm-cm. The suspension is agitated to promote the extraction of soluble salts. An aliquot of the suspension is filtered and used to determine the anion and cation concentrations by ion chromatography. This test can also be used for testing nonhydraulic injection grouts.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

In this method, hardened injection grout is ground in a ceramic ball mill to obtain a maximum grain size of 0.5 mm, instead of being crushed with a hammer to a maximum grain size of 2 mm, as specified by Bläuer Böhm. The finer particle size increases the rate of extraction, and the use of a ball mill provides consistent particle size distributions when compared with manual crushing. Similar to lime mortars, a 1:100 sample weight to water volume ratio is used, since aliquots of these solutions can be tested without dilution. The duration of agitation and interval of testing are increased from 2 minutes to at least 24 hours, in order to also extract salts that have a low solubility at room temperature (23°C ± 2°C). While determining the soluble salt content of injection grouts after a 24 hour agitation is found to provide a general idea of the soluble salt content, it is highly recommended that measurements be repeated after 30 days of agitation, in order to determine the concentrations of ions such as sulfate. This procedure is explained based on the equipment used at the GCI. Other similar equipment can also be used.

### Equipment and Materials

- ceramic ball or mixer mill (e.g., Retsch Laboratory mixer mill MM 2000 with a 10 mL zirconium grinding jar and one 12 mm zirconium ball, and capable of up to 1800 $min^{-1}$ vibrational frequency)
- balance accurate to within 0.0001 g
- flat laboratory shaker (e.g., IKA Labortechnik KS 250 Basic) with a speed up to 550 $min^{-1}$
- 1 mL syringe
- 15 mL plastic container with screw-on lid or centrifuge tube
- ultrapure water (resistivity no greater than 18.2 Mohm-cm)
- ion chromatography filter with 0.2 µm pore size (13 mm polyethersulfone [PES] membrane)
- 1 mL polypropylene vial with a polyethylene snap cap
- oven
- desiccator and silica gel
- mechanical pipette with 10 mL capacity
- thermometer accurate to within 1°C
- RH meter accurate to within 1%
- sieve with 0.5 mm mesh opening
- ion chromatography instrument (e.g., Dionex DX-500 chromatograph)

### Specimen Preparation

1. Grind the hardened grout, which is at least 4 weeks sealed-cured (i.e., not allowed to dry, kept in sealed containers, as for splitting tensile test specimens in columns) in a ceramic ball or mixer mill for 5 minutes at around 1800 $min^{-1}$ to achieve a maximum grain size of 0.5 mm.
2. Dry ground grout in an oven at 40°± 1°C for 20 hours and cool the grout to ambient temperature in a desiccator for 4 hours.
3. Sieve the oven-dried grout and collect the material passing through the 0.5 mm mesh opening. Continue to dry the collected material in the oven at 40°± 1°C for 20 hours and cool the grout to ambient temperature in a desiccator for 4 hours before weighing. Repeat the weighing and drying procedure until a constant weight is reached; this is achieved when the difference between two successive measurements at a 24-hour interval is less than or equal to 0.1%. Drying of the specimen is preferred before conditioning since the grout reaches stable room conditions much faster as a result of sorption and desorption hysteresis.

4. Store the grout in a desiccator with silica gel at 23°C ± 2°C for 4 hours before sampling. Conditioning to normal room conditions is an important step before extraction, since solubility will be affected by the temperature of the specimen. The RH during conditioning should be kept at less than 70% to avoid water uptake by possible hygroscopic salts in the grout.

### Extraction

1. 0.1 g of grout is weighed on an analytical balance to four decimal places and poured into a sterile 15 mL plastic container.
2. 10 mL of ultrapure water (resistivity no greater than 18.2 Mohm-cm) is pipetted into the plastic container, and the lid is tightly closed.
3. The grout and water mixture is shaken on a flat laboratory shaker at a rate of 200 $min^{-1}$ for at least 24 hours at room temperature (23°C ± 2°C).
4. An aliquot of the suspension is removed with a 1 mL syringe and filtered through a 0.2 µm ion chromatography filter into a 1 mL polypropylene vial with a snap cap.

### Procedure

An example procedure based on the Dionex DX 500 Chromatograph, used at the GCI, is as follows:

1. Quantitative analysis of inorganic soluble salts is performed on a Dionex DX 500 chromatograph, which consists of an AS3500 autosampler with a 100 µL sample loop, GP40 pump, ED40 conductivity detector, and LC20 column compartment fitted with an ASRS-300 4 mm anion self-regenerating suppressor or a CSRS-300 4 mm cation self-regenerating suppressor. The ions required for suppression are made by the continuous electrolysis of water within the suppressors at a current setting of 100 mA. The result is a suppression of the eluent conductivity and amplification of the analyte signal.
2. Sodium, potassium, calcium, and magnesium ion concentrations are determined with an IonPac #CS12A Analytical 4 × 250 mm column coupled with a CSRS-300 4 mm cation self-regenerating suppressor. The eluent is 22 mM of $H_2SO_4$, with a flow rate of 1.0 mL/min.
3. Chloride, nitrite, nitrate, and sulfate ion concentrations are determined by ion chromatography using an IonPac AS12A Analytical Column coupled with an ASRS-300 4 mm anion self-regenerating suppressor. The eluent is 1.8/1.7 mM carbonate/bicarbonate, with a flow rate of 1.5 mL/min.
4. A standard Dionex mixture of cations and anions is used for calibration.

### Results

1. Data acquisition, peak analysis, and calibration curves are mainly generated using software provided with the instrument. The ion concentrations are determined in milligrams per liter ($mg \cdot L^{-1}$) by comparison of the peak heights or areas noted for the ions in the specimen to the prepared calibration curves.
2. Ion concentrations are commonly expressed in terms of milliequivalent per gram (meq/g) or milliequivalent per 100 grams (meq/100 g) of hardened grout.

### Note

1. Christine Bläuer Böhm, Assessment of quantitative salt analysis by the water extraction method on lime mortars, in *Proceedings of the 8th International Congress on Deterioration and Conservation of Stone, 30 Sept.–4 October 1996, Berlin,* ed. Josef Riederer (Berlin: Möller Druck und Verlag, 1996), 1505–19.

## 2.7. Capillary Water Absorption

### Aim

This test is used to determine the water absorption behavior of hardened grout by a gravimetric method. The absorption of water in a hardened grout should correspond to that of the materials being grouted to ensure compatibility of original and intervention materials.

**Figure 2.7.1** Example of a commercially available injectability apparatus with an empty column.

### Description

The method uses the procedure of RILEM test number II.6—Water Absorption Coefficient (Capillarity)—and it uses the sample size requirements of NORMAL 11/85—Capillary Water Absorption and Capillary Absorption Coefficient. Samples are prepared using the equipment described in EN 1771—Determination of Injectability Using the Sand Column Test. An apparatus (figs. 2.7.1 and 2.7.2) is used to fill an empty transparent plastic

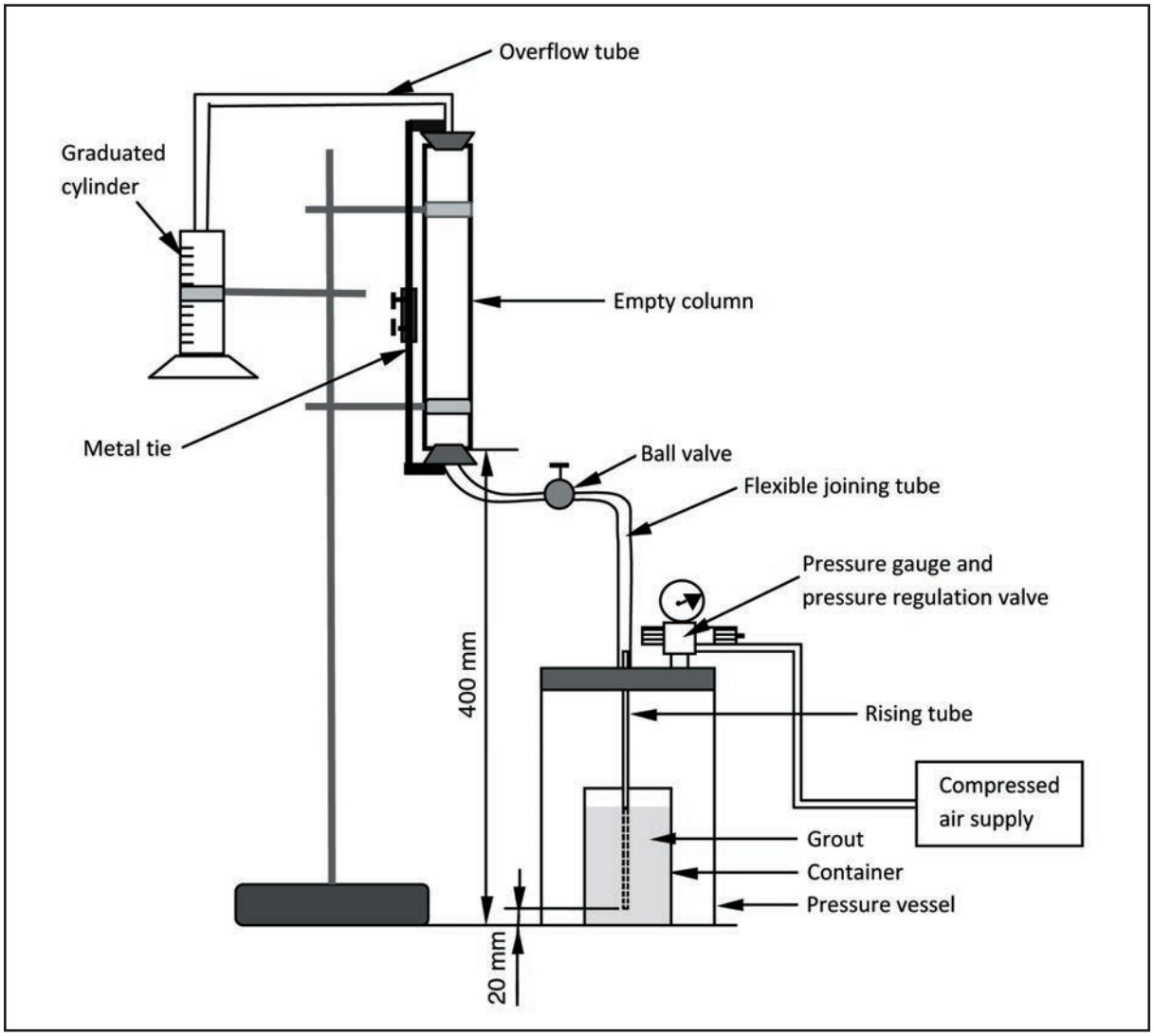



**Figure 2.7.2** Simplified drawing of injectability apparatus (details can be found in EN 1771).

column with grout under constant pressure. The column is held vertically, and grout is injected from the bottom of the tube. The grout specimens are removed from the column with a tile saw after curing is complete. They are dried in an oven and placed on a perforated stand located in a water-filled tray. The weight change of the specimen is used to calculate the amount of water absorbed.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

The selected shape (cylindrical) and size (22.00 ± 0.05 mm in diameter and 100 ± 1 mm in height) of the grout specimen and the relatively low drying temperature (40°C ± 1°C) in this method are designed to minimize specimen cracking during curing, demolding, and drying. The specified dimensions are also suitable for testing brittle and heterogeneous injection grouts by providing a large enough sample volume and a surface area to volume ratio of 2.0 (NORMAL 11/85). The challenge of placing and compacting the grout into smaller-diameter individual molds is resolved by injecting the grout into a plastic tube long enough to obtain three specimens after sectioning with a tile saw.

### Notes

All materials, the test apparatus, and columns are stored under the same conditions (23°C ± 2°C and 60% ± 10% RH) at which the test will be run.

### Equipment and Materials

- injectability apparatus from EN 1771 (figs. 2.7.1 and 2.7.2)
- laboratory stand
- metal tie (fig. 2.7.3) or fabric strap to hold the two perforated stoppers attached to the column in place during specimen preparation (the metal tie used at the GCI consists of two aluminum rods, two U-shaped aluminum plates screwed to one end of each rod, and a short aluminum tube connecting the rods; the aluminum tube has two drilled holes to insert two small butterfly screws to secure the position of the rods)
- transparent rigid plastic (e.g., polymethyl methacrylate, PMMA) tube with 22.00 ± 0.05 mm inner diameter and around 1.75 mm wall thickness; the length of the tube used is 1.82 m
- two solid rubber stoppers (size 4)
- metal or plastic cylindrical container approximately 80 mm in diameter and 175 mm high
- 25 mL graduated glass cylinder

**Figure 2.7.3** Example of a metal tie for the injectability apparatus.

- adhesive tape
- oven
- desiccator and desiccant (e.g., silica gel)
- caliper with a resolution of 0.01 mm
- balance accurate to within 0.01 g
- tray with a surface area more than 20 times larger than the inflow surface area of the specimen
- deionized water
- perforated stainless steel metal or plastic stand
- lint-free cloth
- tile saw with a PMMA cutting blade
- V-shaped specimen holder for cutting
- thermometer accurate to within 1°C
- RH meter accurate to within 1%
- stopwatch
- transparent plastic box, large enough to provide cover over the water-filled tray and the upright specimen
- 4 mil plastic sheet (e.g., window insulation sheet) to cover the open side of the plastic box

### Column Preparation

1. Cut transparent rigid plastic tube into columns 320 mm in length using a tile saw.

### Specimen Preparation

1. Place the empty column on the stand. First attach the bottom rubber stopper connected to the flexible joining tube and then the top one connected to the overflow tube (fig. 2.7.2).
2. Secure the column and the stoppers with the metal tie or fabric strap (fig. 2.7.2 and fig. 2.7.3).
3. Prepare the grout and pour 500 g of the grout into the test container.
4. Place the container in the pressure vessel (fig. 2.7.2) and close the lid of the vessel.
5. Turn on the compressed air supply with the ball valve on the flexible tube closed; adjust the pressure in the vessel to 0.05 MPa (fig. 2.7.2), a lower pressure than that used in the injectability test, since there is no back pressure created by granular material.
6. Check the vessel for leaks: If the pressure in the vessel decreases, release the pressure, open the vessel lid, reclose it, and repeat step 5.
7. Open the ball valve on the flexible tube (fig. 2.7.2). The experiment should begin within 4 minutes after the completion of mixing.
8. Continue injection until the column is full.
9. Close the valve on the flexible tube and release the pressure in the vessel.
10. Remove the column from the stand and seal the column using two solid rubber stoppers and adhesive tape: First place the top solid rubber stopper and tape it, then turn the column upside down and place the second solid rubber stopper and tape it (fig. 2.7.4).
11. Store the column vertically, ensuring that the bottom of the column during injection remains the bottom during storage.
12. Cure the column under sealed conditions at 23°C ± 2°C for 4 weeks.
13. Saw the column perpendicular to its axis to obtain 3 cylindrical specimens 100 ± 1 mm long.
14. While the specimen is held and positioned using a V-shaped holder, extrude each specimen from the plastic tube by sawing the plastic tube covering the cylindrical specimen along the length without making any saw marks on the specimen (fig. 2.7.5) or simply by pushing the specimen with a finger from one end when possible.
15. Record the position of each specimen in the column.

### Procedure

1. Dry the specimens in an oven at 40°C ± 1°C for 20 hours, and cool the specimens to ambient temperature in a desiccator for 4 hours before weighing.

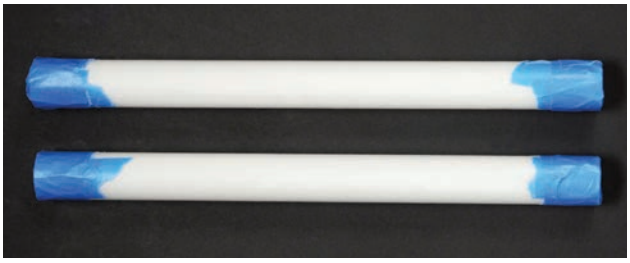


**Figure 2.7.4** Columns filled with grout.

**Figure 2.7.5** Slitting of a cylindrical plastic tube with the tile saw. The specimen is held and positioned under the saw by means of a V-shaped holder.

   Repeat the drying and weighing procedure until a constant weight is reached; this condition is achieved when the difference between two successive measurements, at a 24 hour interval, is less than or equal to 0.1% of the weight of the specimen.
2. Measure the length (l) and diameter (d) of each specimen to the nearest 0.1 mm, using the caliper.
3. Weigh the dry specimen to the nearest 0.01 g ($M_0$).
4. Fill the tray with deionized water until the water level is 2 mm above the perforated stand. During testing, the water level in the tray is kept constant by the addition of water as needed.
5. Place the specimen on the stand and start the stopwatch (fig. 2.7.6).

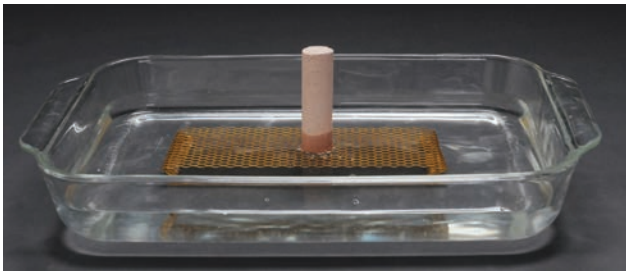


**Figure 2.7.6** Specimen absorbing water after being placed in a water-filled tray. The level of water absorbed is indicated by the darker area at the bottom of the grout specimen.

6. Stop the stopwatch after 30 seconds and simultaneously remove the specimen from the stand. Lightly blot the wet face with a damp cloth to remove surface water and weigh the specimen ($M_t$) while the wet face is at the top.
7. Place the specimen back on the stand as quickly as possible; start the stopwatch again.
8. Repeat the weighing procedure at 1, 2, 5, 10, 15, 30, and 60 minutes, and then every hour until the water level reaches the top of the specimen or until the difference between two successive measurements is less than or equal to 1%.
9. The test setup is covered with a plastic box and sheeting to minimize evaporation and to control the RH of the surroundings (fig. 2.7.7). Desiccant can be used to prevent condensation.

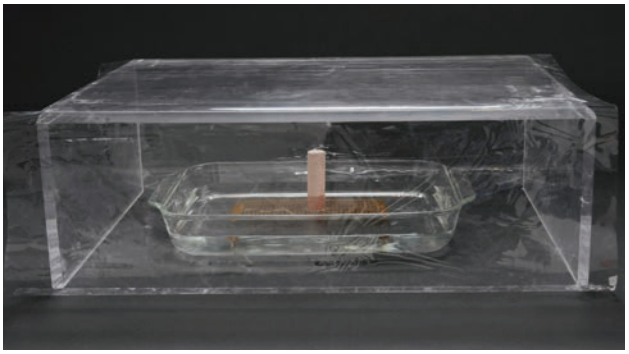


**Figure 2.7.7** Covered test setup. The plastic box prevents evaporation of the water in the tray. The box is placed on its side, and its open end is covered by a plastic sheet.

## Results

1. The amount of absorbed water after time t ($\Delta M_t$), in grams, is calculated as follows:

$$\Delta M_t = M_t - M_0 \qquad (2.7.1)$$

where $M_t$ is the weight of the specimen at time $t$ in grams, and $M_0$ is the dry weight of the specimen at $t = 0$ in grams.

2. The weight of water absorbed per unit area (m) in kilograms per square meter ($kg \cdot m^{-2}$) is calculated as follows:

$$m = \frac{\Delta M_t}{\pi \times \left(\frac{d}{2}\right)^2} \times 10^3 \qquad (2.7.2)$$

where $\Delta M_t$ is the amount of water absorbed after time $t$, in grams, and $d$ is the diameter of the grout specimen in millimeters.

3. The weight of water absorbed per unit area (m) is plotted as a function of the square root of time in seconds ($\sqrt{t}$). The slope of the straight part of this curve is the water absorption coefficient, A, in kilograms per square meter per square root of seconds ($kg \cdot m^{-2} \cdot s^{-1/2}$) (fig. 2.7.8).
4. Calculate the average water absorption coefficient using the results of three specimens. Discard any individual result if the average and the individual result differ more than 10% from each other. Average water absorption coefficient should be obtained from at least two individual results; otherwise, the test should be repeated.
5. An example data collection sheet is given in fig. 2.7.9.

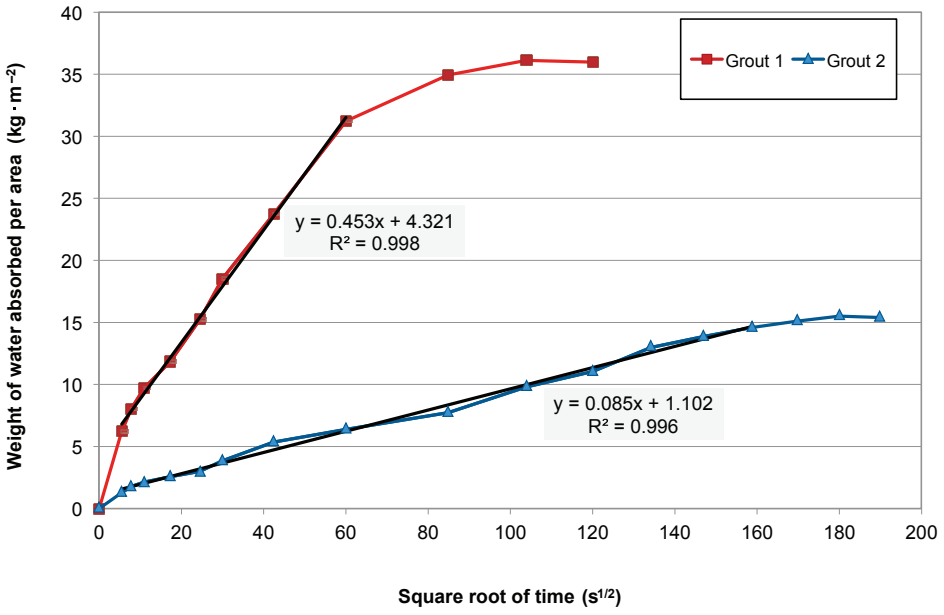



**Figure 2.7.8** Examples of capillary water absorption curves of two injection grouts: The water absorption coefficient of grout 1 (red) and grout 2 (blue) are obtained from the slope of the straight part of the curves as 0.453 $kg \cdot m^{-2} \cdot s^{-1/2}$ and 0.085 $kg \cdot m^{-2} \cdot s^{-1/2}$, respectively (m = weight of water absorbed per unit area; $R^2$ = coefficient of determination).

## CAPILLARY WATER ABSORPTION

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |

**l (mm):** Length of the specimen
**d (mm):** Diameter of the specimen
**$M_0$ (g):** Dry weight of the specimen at time t = 0
**t (s):** Time
**$M_t$ (g):** Weight of the specimen at time t
**$\Delta M_t$ (g):** Weight of absorbed water after time t
**m (kg·m$^{-2}$):** Weight of absorbed water per unit area

**EQUATIONS**

| | |
|---|---|
| $\Delta M_t = M_t - M_0$ | $m = \frac{\Delta M_t}{\pi \times \left(\frac{d}{2}\right)^2} \times 10^3$ |

| Specimen no. | l (mm) | d (mm) | $M_0$ (g) |
|---|---|---|---|
| 1 | | | |
| 2 | | | |
| 3 | | | |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Specimen 1 | t (s) | | | | | | | | | | |
| | $M_t$ (g) | | | | | | | | | | |
| | $\Delta M_t$ (g) | | | | | | | | | | |
| | m (kg·m$^{-2}$) | | | | | | | | | | |
| | t (s) | | | | | | | | | | |
| | $M_t$ (g) | | | | | | | | | | |
| | $\Delta M_t$ (g) | | | | | | | | | | |
| | m (kg·m$^{-2}$) | | | | | | | | | | |
| Specimen 2 | t (s) | | | | | | | | | | |
| | $M_t$ (g) | | | | | | | | | | |
| | $\Delta M_t$ (g) | | | | | | | | | | |
| | m (kg·m$^{-2}$) | | | | | | | | | | |
| | t (s) | | | | | | | | | | |
| | $M_t$ (g) | | | | | | | | | | |
| | $\Delta M_t$ (g) | | | | | | | | | | |
| | m (kg·m$^{-2}$) | | | | | | | | | | |
| Specimen 3 | t (s) | | | | | | | | | | |
| | $M_t$ (g) | | | | | | | | | | |
| | $\Delta M_t$ (g) | | | | | | | | | | |
| | m (kg·m$^{-2}$) | | | | | | | | | | |
| | t (s) | | | | | | | | | | |
| | $M_t$ (g) | | | | | | | | | | |
| | $\Delta M_t$ (g) | | | | | | | | | | |
| | m (kg·m$^{-2}$) | | | | | | | | | | |

**Figure 2.7.9** Data collection sheet for the capillary water absorption test method.

## 2.8. Water Vapor Transmission by the Wet Cup Method

### Aim

This test is used to determine the water vapor transmission rate and permeability through hardened injection grouts. The rate of transmission and permeability of water vapor through a grout should correspond to those of the original materials to ensure compatibility and passage of water vapor through original and intervention layers.

### Description

The method uses the sample size requirements of NORMAL 21/85—Water Vapour Permeability—and the procedure of ASTM E 96—Standard Test Methods for Water Vapor Transmission of Materials. A test specimen is sealed to the open mouth of a test cup containing deionized water and placed in a humidity-controlled chamber. Periodic weighing of the cup and specimen determines the rate of water vapor movement through the specimen from the inside of the cup to the controlled atmosphere. The results are used to calculate both the rate of water vapor transmission (WVTR) and water vapor permeability (WVP). The WVTR is the steady water vapor flow in unit time through unit area, normal to specific parallel surfaces, under specific conditions of temperature and humidity at each surface. The WVP is the time rate of water vapor transmission through unit area of a flat specimen of unit thickness induced by unit vapor pressure difference between two specific surfaces, under specified temperature and humidity conditions.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

The selected shape (discs) and size (44.0 ± 0.5 mm in diameter and 10.0 ± 0.5 mm thick) of the grout specimens (smaller than the standard test specimen size) are designed to minimize cracking of the specimens during curing and demolding. Furthermore, the disc shape simplifies the lateral sealing, and the selected dimensions ensure that the specimen is representative of the material being tested. The dimensional requirements for the specimen include the thickness, at least twice the size of the largest aggregate, and the diameter, at least three times the thickness of the specimen (NORMAL 21/85). To avoid cracking, cured specimens are not oven dried. Instead, one specimen is sealed to an empty cup, and the weight of the specimen and cup is used to correct the weighings of the 3 specimens placed on cups partially filled with water.

### Equipment and Materials

- six ring molds cut from rigid plastic tubing (44.0 ± 0.5 mm inner diameter and 10.0 ± 0.5 mm thick) (fig. 2.8.1)
- glass baseplate
- grease (e.g., petroleum jelly, Vaseline, high-vacuum grease, etc.)
- hot glue gun and glue sticks
- mold release agent
- trowel or spatula
- four glass (fig. 2.8.2) or Plexiglas test cups—the inner diameter of the top shelf is 45.5 ± 0.2 mm, the height is 12.0 ± 0.2 mm; the inner diameter of the lower portion of the cup is 35.0 ± 0.2 mm, the height is 31.0 ± 0.2 mm
- 10 mL syringe
- deionized water
- sealing gum or cord (e.g., Permagum sealing cord)
- balance accurate to within 0.01 g
- caliper with a resolution of 0.01 mm
- thermometer accurate to within 1ºC
- RH meter accurate to within 1%

### Sample Preparation

1. Apply a thin film of grease to the base of the plastic mold; place the greased mold on the glass baseplate.
2. Hold the mold down, clean off the grease where hot glue will be used, and attach the bottom of the mold to the baseplate by gluing it around the bottom edge with hot glue.
3. Prepare six molds for each grout.
4. Spray release agent into the mold.
5. Fill the ring molds to the top with grout within 2 minutes of mixing (fig. 2.8.1). Strike off the grout with the edge of a trowel.
6. Store the samples in a moist cabinet at 90% ± 5% RH and 23ºC ± 2ºC for 2 weeks.
7. Remove the grout samples from the cabinet. Carefully remove the hot glue around the mold and

**Figure 2.8.1** Prepared specimens in the molds.

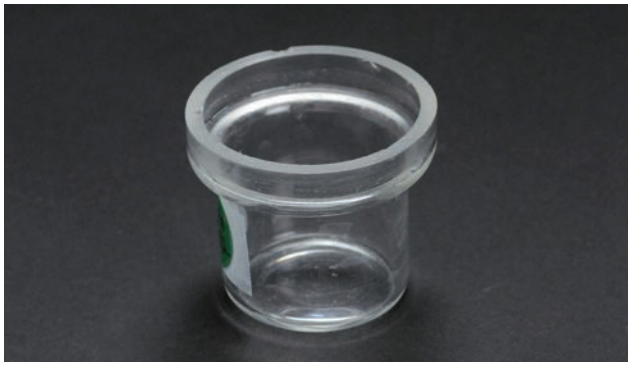


**Figure 2.8.2** Glass specimen cup.

**Figure 2.8.3** Measuring the inner diameter of the lower portion of the cup.

**Figure 2.8.4** Prepared dry cup with specimen.

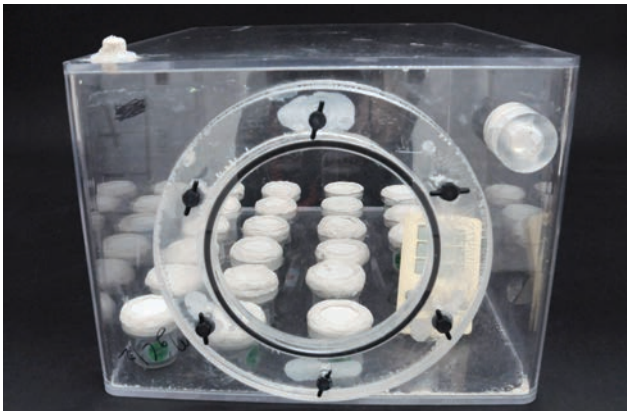


**Figure 2.8.5** Cups in a test chamber.

keep the samples at 65% ± 5% RH and 23°C ± 2°C for 24 hours before demolding.

8. Demold the specimens: First, separate each specimen and mold from the glass plate with a trowel, then push the specimen carefully off the mold.
9. Keep specimens at 65% ± 5% RH and 23°C ± 2°C for another 13 days.
10. Select four specimens out of the six prepared specimens. Selected specimens should not include any visual cracking on the surface, and the top and bottom of each specimen should be parallel.
11. Measure the thickness (δ) of each specimen. The thickness will be measured at two positions perpendicular to each other ($\delta_1$ and $\delta_2$).

### Procedure

1. Label the glass cups, and measure the inner diameter of the lower portion of the cup (d) (fig. 2.8.3).
2. Pour 10 mL deionized water into three of the glass cups, leaving one cup empty.
3. Place the sealing gum inside the rim of the cup (i.e., the top shelf of the cup) and place the specimen on it. Gently push the specimen into the sealing gum, being careful not to apply excessive pressure, so as to avoid cracking the specimen or pushing the sealing gum into the inner diameter of the lower cup.
4. Continue to seal the side and top of the specimen with sealant (fig. 2.8.4), taking care not to cover the area at the top of the specimen corresponding to the mouth area of the lower cup.
5. Weigh the cup and specimen assembly ($M_0$).
6. Place the cups in a test chamber (fig. 2.8.5) and keep the RH at 50% ± 2% and the temperature at 23°C ± 1°C.
7. Weigh the cups (M) every day, until the weight difference between two successive measurements is less than 1%. Record the RH and temperature in the chamber at each weighing.

### Results

1. Calculate the area of the mouth of the cup (a) in square meters with

$$a = \frac{\pi \times d^2}{4 \times 10^6} \tag{2.8.1}$$

where $d$ is the inner diameter of the lower cup in millimeters.

2. Calculate the weight change (ΔM)—loss in this case—of each specimen and cup assembly for each measurement, in grams, with

$$\Delta M = M - M_0 \qquad (2.8.2)$$

where $M$ is the weight of the specimen and cup in grams, and $M_0$ is the initial weight of the specimen and cup, both in grams.

3. The grout specimen used in this method is not dried in an oven to a constant weight because of the risk of cracking. As a result, the weight loss calculated in eq. 2.8.2 is due both to the evaporation of the water in the cup through the specimen and to the drying of the specimen itself. A correction is applied to the weight change to eliminate the effect of specimen drying.

   Correct the weight change per area and thickness of the specimen in the wet cup measured at time $t$, using the weight change per area and thickness of the specimen in the dry cup measured at time $t$, as follows:

$$(\Delta M_{a,\delta})_{corr} = \left(\frac{\Delta M_w \times 10^3}{a_w \times \delta_w}\right) - \left(\frac{\Delta M_d \times 10^3}{a_d \times \delta_d}\right) \qquad (2.8.3)$$

where $(\Delta M_{a,\delta})_{corr}$ is the corrected weight loss per area, and thickness of the specimen in the wet cup at time $t$ in grams per cubic meter ($g \cdot m^{-3}$), $\Delta M_w$ is the weight change of the specimen and wet cup in grams, $a_w$ is the mouth area of the wet cup in square meters, $\delta_w$ is the thickness of the specimen in the wet cup in millimeters, $\Delta M_d$ is the weight loss of the specimen and dry cup in grams, $a_d$ is the mouth area of the corresponding cup in square meters, and $\delta_d$ is the thickness of the specimen in the dry cup in millimeters.

   Multiply the corrected weight change per area and thickness at time $t$—$(\Delta M_{a,\delta})_{corr}$ in grams per cubic meter ($g \cdot m^{-3}$)—and the thickness of the specimen in the corresponding wet cup ($\delta_w$) in millimeters, to obtain the corrected weight change per area of the wet cup, $(\Delta M_a)_{corr}$, as follows:

$$(\Delta M_a)_{corr} = (\Delta M_{a,\delta})_{corr} \times \delta_w \times 10^{-3} \qquad (2.8.4)$$

4. Plot the corrected weight change per area, $(\Delta M_a)_{corr}$, in grams per square meters ($g \cdot m^{-2}$), against time (t) in hours (fig. 2.8.6). The slope of the linear portion of the curve is the rate of water vapor transmission (WVTR) in grams per hour per square meter ($g \cdot h^{-1} \cdot m^{-2}$). The most common way of expressing WVTR is in grams per 24 hours per square meter ($g \cdot 24h^{-1} \cdot m^{-2}$). For meaningful WVTR values, the thickness of the specimen, the water vapor pressure difference, and the temperature during the measurements should be indicated.
5. Water vapor permeability (WVP) of the grout samples in grams per hour per pascal per meter ($g \cdot h^{-1} \cdot Pa^{-1} \cdot m^{-1}$) is calculated with

$$WVP = \frac{WVTR}{\Delta P} \times \delta \times 10^{-3} \qquad (2.8.5)$$

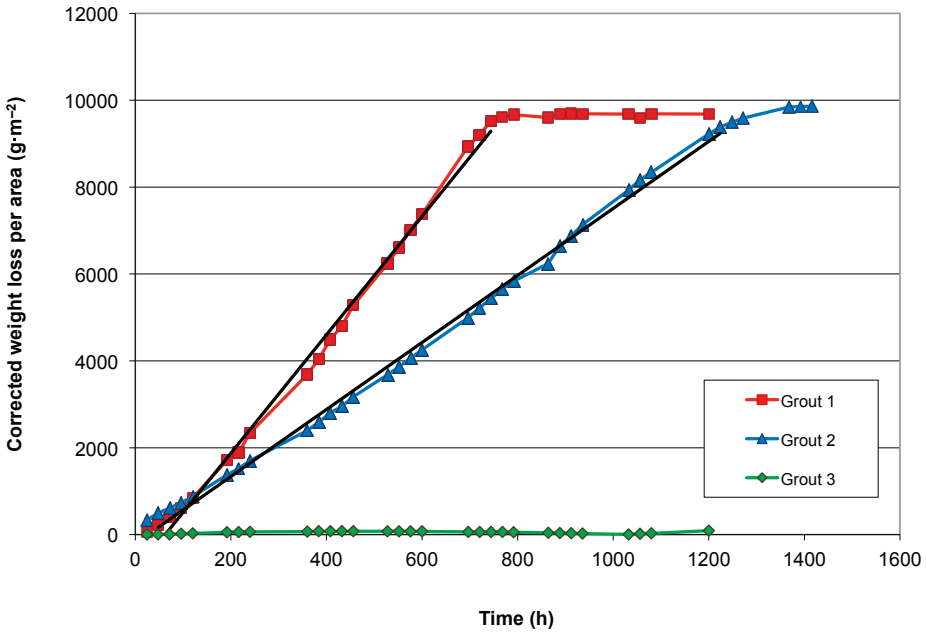



**Figure 2.8.6** Water vapor transmission (WVT) curves of three injection grouts: Rate of water vapor transmission of grout 1 (red line), grout 2 (blue line), and grout 3 (green line) are obtained from the slope of the linear portion of the curves as 12.99 $g \cdot h^{-1} \cdot m^{-2}$, 7.63 $g \cdot h^{-1} \cdot m^{-2}$, and 0.04 $g \cdot h^{-1} \cdot m^{-2}$, respectively. Water vapor permeability (WVP) of grout 1 is $2.0 \times 10^{-5}$ $g \cdot Pa^{-1} \cdot s^{-1} \cdot m^{-1}$; of grout 2.1 $\times 10^{-5}$ $g \cdot Pa^{-1} \cdot s^{-1} \cdot m^{-1}$; of grout $3.6 \times 10^{-8}$ $g \cdot Pa^{-1} \cdot s^{-1} \cdot m^{-1}$.

where δ is the thickness of the specimen in millimeters, and $\Delta P$ is the vapor pressure difference in pascals. The vapor pressure difference is calculated by

$$\Delta P = S_{vp} \times (RH_1 - RH_2) \qquad (2.8.6)$$

where $S_{vp}$ is the saturation vapor pressure at test temperature (2809 Pa at 23°C); $RH_1$ is the relative humidity at the cup (100%), expressed as a fraction (1.00); and $RH_2$ is the relative humidity at the chamber (50%), expressed as a fraction (0.50).

6. Calculate average WVTR and WVP using the results of 3 specimens. Discard any result if the average and the independent result differ more than 10%. Average WVTR and WVP should be obtained by at least 2 individual results; otherwise, the test should be repeated.
7. An example data collection sheet is given in fig. 2.8.7.

## WATER VAPOR TRANSMISSION BY THE WET CUP METHOD

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |

**d (mm):** The inner diameter of the lower portion of the specimen cup
**$\delta_1$ and $\delta_2$ (mm):** Two measurements of thickness of the specimen taken perpendicular to each other
**$\delta$ (mm):** Average thickness of the specimen
**$M_0$ (g):** Initial weight of the specimen and cup
**t:** Time when weighing is done
**M (g):** Weight of the specimen and cup

| Specimen no. | d (mm) | $\delta_1$ (mm) | $\delta_2$ (mm) | $\delta$ (mm) | $M_0$ (g) |
|---|---|---|---|---|---|
| 1 (dry cup) | | | | | |
| 2 | | | | | |
| 3 | | | | | |
| 4 | | | | | |

| Time (t) | | | Temp. | RH | M (g) | | | |
|---|---|---|---|---|---|---|---|---|
| Date | Hour | Min | (°C) | (%) | 1 (dry cup) | 2 | 3 | 4 |
| | | | | | | | | |
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**Figure 2.8.7** Data collection sheet for the water vapor transmission test method.

# 3. Supplementary Laboratory Testing Procedures



## 3.1. Flow with Injectability Columns

### Aim

The aim of this test is to determine the ability of a grout to flow through a capillary network of different granular medium, either dry or prewetted, under gravity. This test is recommended when gravity-based injection grouting will be used (e.g., for floor mosaics). Depending on the type of voids, cracks, and delaminations to be grouted, grouts that exhibit greater or lesser flow might be desirable.

### Description

This test uses the concept of measuring the flow of injection grouts similar to the concept of the flow cone test (Marsh cone, ASTM C 939, EN 445, etc.), which determines the time of efflux of a specified volume of grout in a standardized cone passing through a nozzle under gravity, and adapts it to injection grouts for architectural surfaces where the grout flows through interconnected cracks, which are simulated by a capillary network created by granular material (EN 1771—Determination of Injectability Using the Sand Column Test). The grout is poured into a column that consists of a transparent plastic tube half filled with granular material (fig. 3.1.1). The column is held vertically, and a constant volume of grout is added from the top of the tube. The distance of penetration of the grout and the corresponding time are used to plot a flow curve and to classify the ease of flow. This test can also be used for testing nonhydraulic injection grouts.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

Columns of differing granular material are prepared following the procedure of EN 1771— Determination of Injectability Using the Sand Column Test—with the exception that the granular material is filled to half of the column height (fig. 3.1.1). Silica sand (EN 196-1) specified in the standard test protocol is replaced by crushed granular material that is more representative of materials used in archaeological and historic architectural systems. At the GCI, crushed brick and crushed travertine are used for this purpose. The reason for this modification is the low absorption capacity of EN 196-1 silica sand (3% by weight) and the fact that it does not represent conditions in most walls. Crushed brick and crushed travertine, typical of architectural substrates for plasters, wall paintings, and mosaics, are used because they represent high and low water absorptive media, respectively. The amount of water absorbed after a 10 minute soaking time is approximately 30% by weight for crushed brick and 12% by weight for crushed travertine. It is suggested that testing be carried out with both media when the absorption conditions of the area where the grout will be used are not known.

Sand gradation specified in the standard, which simulates resistance to flow into a 0.2 mm crack in concrete, is replaced by granular material (e.g., crushed brick or crushed travertine) with grain sizes between 2 and 4 mm, simulating a crack width of approximately 0.3–0.6 mm. The particle size distributions of the crushed brick and crushed travertine, as determined by sieve analysis, are given in table 3.1.1, and the comparison of the particle size distributions of the granular materials used in this test method with the particle size distribution of silica sand (EN 196-1) is given in figure 3.1.2.

**Table 3.1.1** Particle size distribution of the granular material used in this test method.

| Sieve size (mm) | Cumulative passing percentage (%) |
|---|---|
| 4.00 | 100 |
| 3.35 | 84 |
| 2.36 | 34 |
| 2.00 | 0 |

### Note

All materials and columns are stored under the same conditions (23°C ± 2°C and 60% ± 10% RH) at which the test will be run.

### Equipment and Materials

- transparent rigid plastic tube (e.g., PMMA) with 22.00 ± 0.05 mm inner diameter and approximately 1.75 mm wall thickness; the length of the tube used is 1.82 m
- 2–4 mm grain size crushed brick (71 ± 2 g per column) and/or crushed travertine (103 ± 2 g per column); see table 3.1.1 for gradation details
- two 22 mm diameter stainless steel wire mesh discs with 0.5 mm mesh opening per column
- two solid rubber stoppers (size 4) per column
- clamp stand
- plastic or wooden rod at least 200 mm long with a diameter between 15 and 20 mm, to place the top wire mesh disc in place
- 100 mL beaker
- 100 mL glass graduated cylinder
- two 250 mL beakers
- 200 mm ruler in millimeters
- balance accurate to within 0.1 g
- 200 mL deionized water or water and alcohol, per column, if prewetting will be applied
- stopwatch
- permanent marker
- thermometer accurate to within 1ºC
- RH meter accurate to within 1%
- tile saw equipped with a PMMA cutting blade
- dessicator and dessicant (e.g., silica gel)
- oven

### Column Preparation

1. Dry the granular materials in an oven at 105ºC for 20 hours, and cool the specimens to ambient temperature in a desiccator for 4 hours before weighing. Repeat the drying and weighing procedure until a constant weight is reached, which is achieved when the difference between two successive measurements at a 24 hour interval is less than or equal to 0.1%.
2. With a tile saw, cut transparent plastic tubes (22.00 ± 0.05 mm inner diameter) into columns 390 mm in length. At least two columns per grout are needed for each media and injection condition.
3. Place a wire mesh disc and a rubber stopper in the lower end of the column. The wire mesh should fit tightly into the tube to prevent any movement of granular material once the rubber stopper is removed.
4. Mark the columns at 30 mm intervals, starting at the wire mesh level, up to a height of 180 mm.
5. Weigh each column with the second wire mesh disc and two stoppers to the nearest 0.1 g ($M_l$). Skip this step when the weight of granular material needed to prepare a column has already been determined.

**Figure 3.1.1** Columns half filled with crushed brick (left) and crushed travertine (right).

6. If known, weigh the granular material ($M_f$) needed for each column: 71 ± 2 g for a crushed brick column, and 103 ± 2 g for a crushed travertine column.
7. Fill the column with crushed brick or crushed travertine in two equal layers of 90 ± 5 mm in height (fig. 3.1.1).
8. After filling the first layer, apply 50 lateral shocks by slowly hitting the vertically held column against the edge of a table while holding it from the bottom end. Shocks should be evenly distributed over the height of the layer (10 times from the bottom to the top of the layer), and the column should be rotated (5 rotations).
9. Repeat step 8 after filling the second layer.
10. Check the total height of the column. The total height of the compacted crushed brick or crushed travertine in the column should be 180 ± 1 mm. If it is not, empty the column and repeat the filling procedure. The compaction of the medium is very important in order to obtain similar capillary networks.
11. Push the second wire mesh disc using the plastic rod until it touches the top of the granular material. Be careful not to push the granular material; pushing would lead to additional compaction.

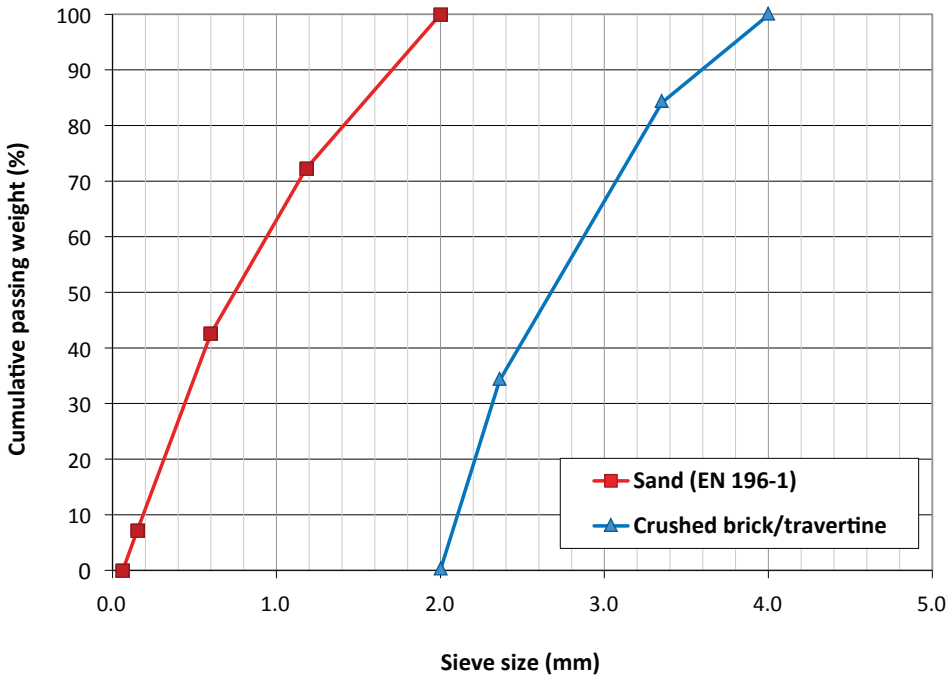



**Figure 3.1.2** Particle size distribution curves of sand (EN 196-1), and crushed brick and crushed travertine.

12. Place the second stopper into the upper end of the column.
13. Weigh the column half filled with granular material to the nearest 0.1 g ($M_2$), keeping it in an upright vertical position. Skip this step when the weight of granular material needed to prepare a column has already been determined.
14. Store the columns upright vertically until testing is performed.

### Procedure

1. Place the column vertically in a clamp stand (fig. 3.1.1), and place 100 mL graduated cylinder under the column (fig. 3.1.3).
2. Carefully remove the rubber stoppers.
3. Prepare the grout, measure 70 mL grout in the 100 mL beaker, and quickly pour the grout into the column.
4. Start the stopwatch when the grout reaches the granular material (0 mm mark line).
5. Note the time elapsed for the grout to reach the 30 mm, 60 mm, 90 mm, 120 mm, 150 mm, and 180 mm (wire mesh level) marked lines.
6. Continue until the column is filled and 5 mL excess grout is collected at 1 mL/min in the graduated cylinder (fig. 3.1.3), or until the penetration of grout in the column is slower than 10 mm/min.
7. Stop the stopwatch when one of the conditions given in step 6 is fulfilled (fig. 3.1.3).

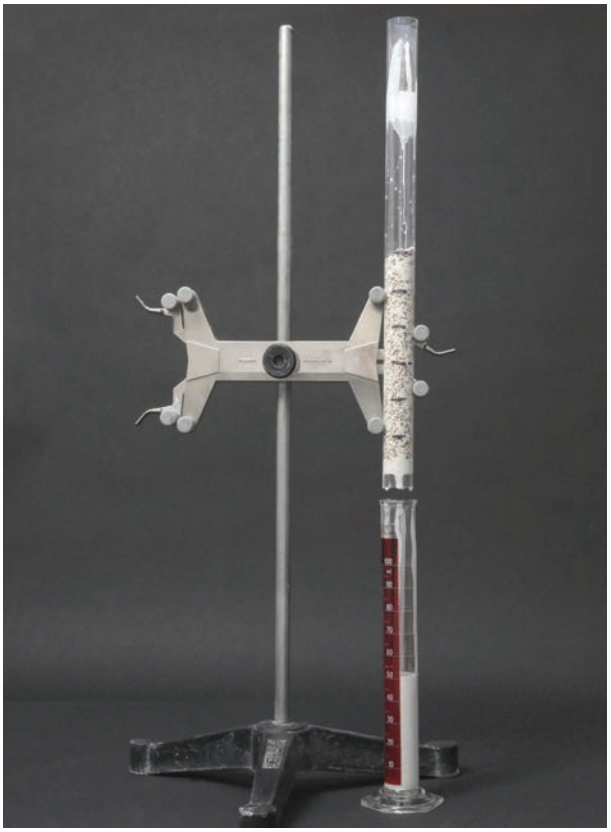


**Figure 3.1.3** A column half filled with crushed travertine after grout has been poured into it. This is an example of an easy (E) flow, with over 5 mL excess grout collected in the graduated cylinder.

### Prewetting

Prewetting of the granular absorbent medium before injecting grouts is a common practice. Prewetting can be useful to avoid rapid drying and ensure proper adhesion. The following steps explain how to prewet the columns to investigate the effects of prewetting on flow:

1. Place the column half filled with granular material vertically in a clamp stand (fig. 3.1.1) and place a 250 mL beaker under the column.
2. Carefully remove the rubber stoppers.
3. Measure 200 mL deionized water using a 250 mL beaker, and slowly pour the water into the column before adding the grout. Let the excess water drain for 10 minutes.
4. Place graduated cylinder under the column and follow the procedural steps from 3 through 7.

### Results

1. Calculate the weight of dry crushed brick or crushed travertine ($M_f$) in the column in grams,

$$M_f = M_2 - M_1 \qquad (3.1.1)$$

where $M_1$ is the weight of the empty column with two wire mesh discs and stoppers, and $M_2$ is the weight of the column half filled with granular material and two stoppers, both in grams.

The weight of crushed brick or crushed travertine used to fill the column is an important parameter that indicates the degree of compaction of fill material in each column. The weight of granular material needed to prepare a 180 ± 1 mm column should be determined for each medium type, and the same amount of granular material should be used for the preparation of each column. These values are determined as 71 ± 2 g for crushed brick and 103 ± 2 g for crushed travertine used at the GCI. When other granular material is used during column preparation, the amount of fill in the column should be calculated with equation 3.1.1.

2. Draw flow curves for each grout by plotting the distance reached in the column by the grout along the y-axis and the measured corresponding time along the x-axis:

$$h = func(t) \qquad (3.1.2)$$

where $h$ is the depth reached by the grout in the column in millimeters, and $t$ is the time, in seconds, taken by the grout to reach the differential reference marks drawn on the column. The flow of the grout can be classified as:

a. Easy (E)—if the grout reaches the bottom of the column and 5 mL of excess grout is collected at 1mL/min;
b. Feasible (F)—if the grout reaches the bottom of the column but the 1 mL/min overflow is not achieved;

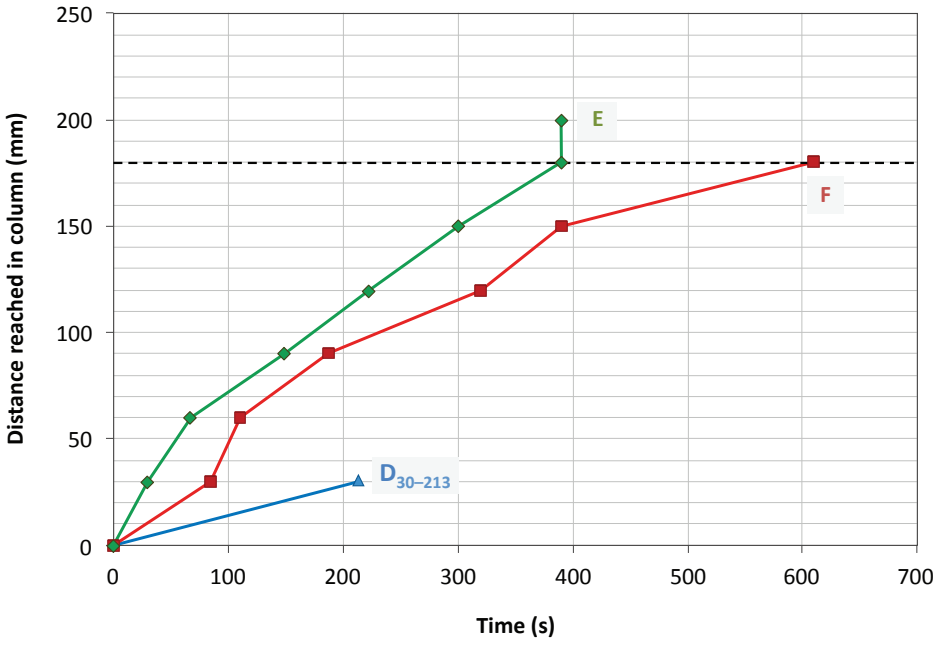



**Figure 3.1.4** Flow curves of three grouts in a prewetted crushed travertine column. The total height of the column filled with granular media is 180 mm—indicated as a black dashed horizontal line on the plot. Overflow, in the case of easy (E) flow (green line), is indicated by an extra data point at 200 mm. The grout with feasible (F) flow, is shown in red. In the case of difficult flow (D) (blue line), the depth reached in the column (h) in millimeters and the corresponding time (t) in seconds are indicated as $D_{30-213}$.

c. Difficult (D)—if the grout flow is halted or the grout penetration is slower than 10 mm/min before reaching the bottom of the column. Record the depth reached in the column (h) in mm and the corresponding time (t) in seconds ($D_{h-t}$).

As an example, flow curves for three different grouts, all injected into prewetted crushed travertine columns, are plotted in figure 3.1.4, and the appropriate category for each grout is indicated on the graph.

3. Repeat the test for each condition until two experimental runs provide the same classification. For grouts classified as difficult, the average depth and time of the two runs are used as the final values. The average depth reached in the column of the two runs should not differ more than 25% from each measured depth; otherwise, the test should be repeated.
4. An example data collection sheet is given in figure 3.1.5.

## FLOW WITH INJECTABILITY COLUMNS

| | | | | |
|---|---|---|---|---|
| **Grout name** | | **Room** | **Temperature (°C)** | |
| **Grout proportions** | | | **RH (%)** | |
| **Operator** | | **Date** | | |

**$M_1$ (g):** Weight of the empty column with wire mesh discs and stoppers
**$M_2$ (g):** Weight of the column filled with crushed brick/travertine, wire mesh discs and stoppers
**$M_f$ (g):** Weight of the crushed brick/travertine in column, calculated using [$M_2 - M_1$]

| | Travertine-wet | | Travertine-dry | | Brick-wet | | Brick-dry | |
|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| **$M_1$ (g)** | | | | | | | | |
| **$M_2$ (g)** | | | | | | | | |
| **$M_f$ (g)** | | | | | | | | |

**h (mm):** Depth of the grout in column
**t (s):** Time taken by the grout to reach the differential reference marks drawn along the column

| | Travertine-wet | | Travertine-dry | | Brick-wet | | Brick-dry | |
|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| **h (mm)** | t (s) | t (s) | t (s) | t (s) | t (s) | t (s) | t (s) | t (s) |
| **30** | | | | | | | | |
| **60** | | | | | | | | |
| **90** | | | | | | | | |
| **120** | | | | | | | | |
| **150** | | | | | | | | |
| **180** | | | | | | | | |
| | | | | | | | | |

**EASY (E):** If the grout reaches the bottom of the column and 5 mL of excess grout is collected at 1 mL/min
**FEASIBLE (F):** If the grout reaches the bottom of the column but the 1 mL/min overflow is not achieved
**DIFFICULT (D):** If the grout flow is halted or slower than 10 mm/min before reaching the bottom of the column; record the depth and time ($D_{h-t}$)

| | Travertine-wet | | Travertine-dry | | Brick-wet | | Brick-dry | |
|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| **Classification** | | | | | | | | |

**Figure 3.1.5** Data collection sheet for the flow with injectability columns test method.

## 3.2. Water Retention and Release

### Aim

This test is used to determine the ability of a grout to retain water when subjected to suction—that is, the test simulates the action of absorptive adjacent porous building materials. The ability of a grout to retain water is important for ease of injectability and flow with reduced separation of liquid and solid phases, proper hydraulic set, reduced shrinkage, and reduced salt migration into original materials.

### Description

The method uses the filtration assembly (figs. 3.2.1 and 3.2.2) and the testing procedure of ASTM C 1506—Standard Test Method for Water Retention of Hydraulic Cement-Based Mortars and Plasters—and the calculations of DIN 18 555 part 7—Testing of Mortars Containing Mineral Binders: Determination of Water Retentivity of Freshly Mixed Mortar by the Filter Plate Method to Determine the Water Retention Value (WRV). Grout is contained in a perforated dish and subjected to controlled vacuum suction for 60 seconds. The water retention value is calculated from the weight difference (water loss) between the grout before and after suction is applied. This method is especially useful when the injectability test cannot be performed. While the method cannot provide all the information obtained from an injectability test, it can help to compare grouts in terms of their ability to be injected and to flow, since the grout with higher water retention capacity will show higher resistance to the absorption of its water by the substrate and therefore will continue to flow farther. Nonhydraulic injection grouts can also be tested using this method.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

This test method follows the ASTM C 1506 testing procedure with two exceptions: (1) The perforated sample dish is not filled to the top edge as required, and instead, a reduced constant volume of injection grout (200 mL) is used. When the sample dish is filled to the top, moving the dish from the balance to the filtration assembly and from the assembly to the balance without losing material presents a challenge for injection grouts, which are less viscous than mortars and plasters; (2) WRV is not calculated from the flow measurements, since the flow table, which is designed to test mortars and plasters, is not appropriate for testing injection grouts, again because of their lower viscosity. Therefore, all calculations are based on weight measurements following DIN 18 555, part 7.

### Notes

Room temperature is maintained at 23°C ± 2°C, and RH of the air is kept at 60% ± 10% during the experiment.


**Figure 3.2.1** Example of a commercially available filtration assembly (water retention apparatus, appendix B).

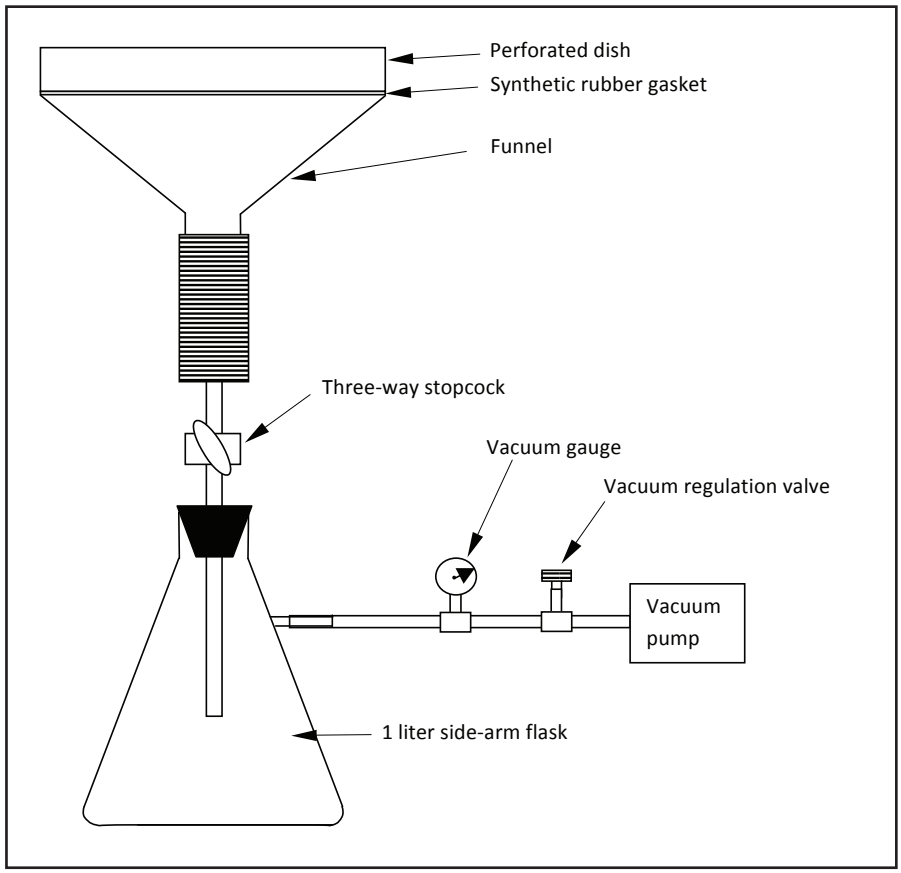



**Figure 3.2.2** Drawing of a filtration assembly (details can be found in ASTM C 1506).

### Equipment and Materials

- filtration assembly (figs. 3.2.1 and 3.2.2)
- hardened, smooth (not rapid) 2.5 µm filter paper with 150 mm diameter (e.g., Whatman 42)
- tamper made from nonabsorptive, nonabrasive, and nonbrittle material with a cross section of approximately 13 × 25 mm and a length of 120–150 mm
- grease (e.g., petroleum jelly, Vaseline, high-vacuum grease, etc.)
- deionized water
- balance accurate to within 0.01 g
- 250 mL glass beaker
- stopwatch
- lint-free cloth
- thermometer accurate to within 1°C
- RH meter accurate to within 1%
- vacuum pump

### Procedure

1. Place a filter paper, prewetted with deionized water and brought to a surface-dry condition, in the perforated dish of the filtration assembly. Weigh the dish and filter paper ($M_1$) to the nearest 0.01 g (fig. 3.2.3).
2. With a beaker, measure 200 mL grout, and slowly pour the grout into the perforated dish. If the grout is not liquid enough to spread evenly across the surface of the dish, tamp 15 times with the tamper. Apply 10 tamping strokes at approximately uniform spacing adjacent to the rim of the dish, with the long axis of the tamping face held at a right angle to the radius of the dish. Apply the remaining 5 tamping strokes at random points distributed over the central area of the dish.

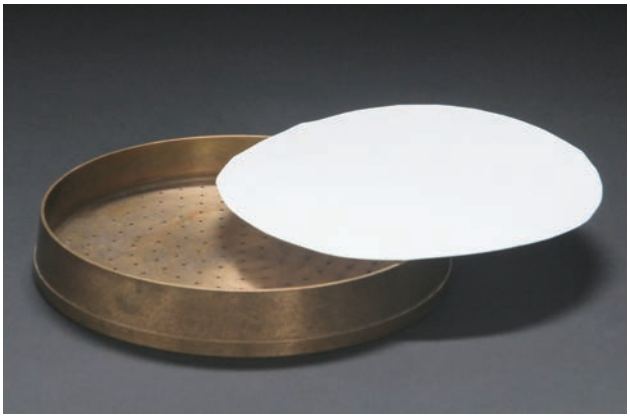


**Figure 3.2.3** Perforated sample dish and filter paper.

3. Weigh the dish filled with grout ($M_2$) to the nearest 0.01 g.
4. Grease the rubber gasket on the rim of the funnel, and place the dish on top of it.
5. Make sure that the three-way stopcock is closed so that vacuum is not applied to the funnel.
6. Adjust the vacuum to 53.00 ± 0.05 millimeters of mercury (mm Hg).
7. Turn the stopcock to apply the vacuum to the funnel, and simultaneously start the stopwatch.
8. After applying suction for 60 seconds, turn the knob on the stopcock to expose the funnel to atmospheric pressure.
9. Remove the dish from the funnel. Quickly dab a damp cloth on the underside of the perforated dish to remove droplets of water, and weigh the dish ($M_3$) to the nearest 0.01 g.

### Results

1. The weight of grout in the perforated dish ($M_g$), in grams, is

$$M_g = M_2 - M_1 \tag{3.2.1}$$

where $M_1$ is the weight of the perforated dish and wet filter paper, and $M_2$ is the weight of the perforated dish, wet filter paper, and grout, all in grams.

2. Water content of the grout in the perforated dish before suction is applied ($W_1$), in grams, is calculated by

$$W_1 = \omega \times M_g \tag{3.2.2}$$

where $M_g$ is the weight of grout in the perforated dish in grams, and ω is the water to grout ratio (by weight), calculated using the grout mix proportions (i.e., total weight of water and dry ingredients). The water to grout ratio, ω, is calculated by

$$\omega = \frac{M_w}{M_w + M_{dg}} \tag{3.2.3}$$

where $M_w$ is the total weight of water in the grout in grams, and $M_{dg}$ is the total weight of dry grout ingredients in grams.

3. The weight of the water extracted by suction ($W_2$), in grams, is given by

$$W_2 = M_3 - M_2 \tag{3.2.4}$$

where $M_3$ is the weight of the perforated dish filled with grout after suction is applied, in grams, and $M_2$ is the weight of the perforated dish and grout before suction is applied, in grams.

4. Water retention value (WRV), to the nearest 1%, is calculated as a percentage using the following formula:

$$WRV = \left(1 - \frac{W_2}{W_1}\right) \times 100 \tag{3.2.5}$$

where $W_1$ is the weight of the water in grout before suction is applied, in grams, and $W_2$ is the amount of water extracted by suction, in grams.

5. The average of two independent measurements is stated as WRV. The average value should not differ more than 10% from each measured value; otherwise, the test should be repeated.
6. An example data collection sheet for this procedure is given in fig. 3.2.4.

## WATER RETENTION AND RELEASE

| | | | | |
|---|---|---|---|---|
| **Grout name** | | **Room** | **Temperature (°C)** | |
| **Grout proportions** | | | **RH (%)** | |
| **Operator** | | **Date** | | |

**$M_w$ (g):** Weight of the water used during grout mixing
**$M_{dg}$ (g):** Total weight of dry grout ingredients used during grout mixing
**ω:** Water to grout weight ratio
**$M_1$ (g):** Weight of perforated dish and wet filter paper
**$M_2$ (g):** Weight of perforated dish and wet filter paper with grout
**$M_g$ (g):** Weight of grout in the perforated dish
**$W_1$ (g):** Water content of the grout before suction
**$M_3$ (g):** Weight of perforated dish and wet filter paper with grout after suction
**$W_2$ (g):** Weight of water extracted by suction
**WRV (%):** Water retention value

### EQUATIONS

| | |
|---|---|
| $\omega = \frac{M_w}{M_w + M_{dg}}$ | $W_2 = M_3 - M_2$ |
| $M_g = M_2 - M_1$ | $WRV = \left(1 - \frac{W_2}{W_1}\right) \times 100$ |
| $W_1 = \omega \times M_g$ | |

| Specimen no. | $M_w$ (g) | $M_{dg}$ (g) | ω |
|---|---|---|---|
| 1 | | | |
| 2 | | | |

from mix proportions

| Specimen no. | $M_1$ (g) | $M_2$ (g) | $M_g$ (g) | $W_1$ (g) | $M_3$ (g) | $W_2$ (g) | WRV (%) |
|---|---|---|---|---|---|---|---|
| 1 | | | | | | | |
| 2 | | | | | | | |

**WRV, average =** ____ %

**Figure 3.2.4** Data collection sheet for the water retention and release test method.

## 3.3. Rheological Measurements

### Aim

This method is used to determine the rheological properties of injection grouts, such as plastic viscosity, apparent yield stress, and thixotropy. Injection grouts are suspensions and may exhibit a variety of rheological properties. The use of additives, such as deflocculents, plasticizers, and others, affects the rheological properties of grouts, by modifying the interaction of particles and fluid, by changing the surface tension of the suspension fluid, and in other ways.

Desirable rheological properties must be assessed on a case-by-case basis, depending on the type of voids, cracks, and delaminations that are to be grouted. As an example, for emergency stabilization of large areas, grouts flowing only when "pushed" under pressure—by syringe—can be used. These thixotropic grouts exhibit a reversible, time-dependent decrease in viscosity depending on applied shear rate.

In general, measurements of rheological properties are not suggested for commercial grouts prepared using proportions (e.g., water content) and mixing procedures recommended by the manufacturer, since the rheological properties of the product have already been optimized. However, these measurements are very useful when designing a new grout with a specific flow behavior or when the manufacturer has failed to supply these measurements for a special application, and when comparing flow characteristics of grouts for a specific use.

### Description

This method loosely follows UNI 11152—Water Suspensions of Hydraulic Binders for Grouting: Characteristics and Test Methods. The measurements are made with a controlled-rate rotational viscometer (fig. 3.3.1) with a Searle-type measuring system combined with a coaxial cylinder. The outer cylinder (cup) is stationary, and the inner cylinder (rotor) is driven by a motor (fig. 3.3.2). The grout fills the space between the two cylinders. The viscous drag friction of the grout against the rotor causes the deflection of a spring, and this is correlated with torque. The sensor system allows the torque data to be mathematically transformed to shear stress and allows the rotor speed to be transformed to shear rate. The characteristics of the cylinders are: $r_i / r_e > 0.9$, and $r_e - r_i > 10 - d_{max}$, where $r_i$ is the radius of the inner cylinder, $r_e$ is the radius of the outer cylinder, and $d_{max}$ is the maximum diameter of the largest particle in the grout, all in millimeters. The results are presented in a flow curve obtained by plotting the shear stress as a function of shear rate, which can also be generated by the viscometer software.

The most common type of flow curve observed for injection grouts is the Bingham plastic (nonideal) curve (fig. 3.3.3). In this case, the differential viscosity approaches a constant value with increasing shear rate, and the differential viscosity in the linear region is called the plastic viscosity ($\eta_{pl}$). When the flow curve is extrapolated from the linear region to the stress axis, the value at which the line crosses the axis is the

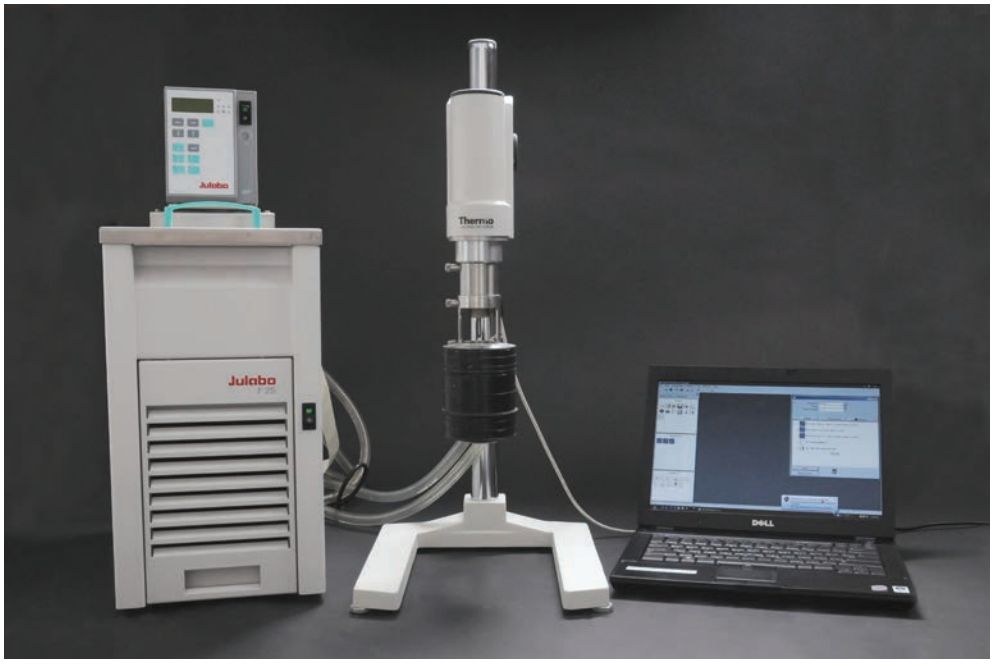



**Figure 3.3.1** A controlled-rate rotational viscometer, Thermo Haake Viscotester VT 550.

**Figure 3.3.2** The inner (left) and outer (right) cylinders of the viscometer.

apparent Bingham yield stress ($\sigma_B^*$). Yield stress is a critical shear stress value below which a viscoplastic material behaves like a solid. Once the yield stress is exceeded, viscoplastic material flows like a liquid. Injection grouts may also exhibit thixotropy. Thixotropic behavior of a grout can be determined by ramping up the shear rate at a constant speed to a maximum value, keeping the shear rate constant for a limited time, and then reducing the shear rate back to the initial shear rate at the same speed. Thixotropic grouts will show a flow hysteresis—i.e., the flow curve obtained while the shear rate is increasing lies above the flow curve obtained while the shear rate is decreasing (fig. 3.3.3).

Nonhydraulic injection grouts can also be tested with this method.

### Notes

All materials are stored at the temperature (23°C ± 2°C) at which the mixing will be conducted and the test will be run. The temperature of the grout is kept constant (23°C ± 2°C) during testing by an external cooling system. The RH of the air is kept at 60% ± 10% during the experiment.

### Equipment and Materials

- rotational viscometer (e.g., Thermo Haake Viscotester VT 550) (fig. 3.3.1)
- coaxial cylinder assembly (fig. 3.3.2)
- external cooling system (e.g., Julabo MP refrigerated circulator) to minimize heating of the grout due to friction and evaporation
- small spatula
- thermometer accurate to within 1°C
- RH meter accurate to within 1%

### Procedure

1. Mix the grout and place it in the viscometer cup. The volume of the grout placed in the cup depends on the diameter of the rotor and the cup, and the viscometer manufacturer's recommendations

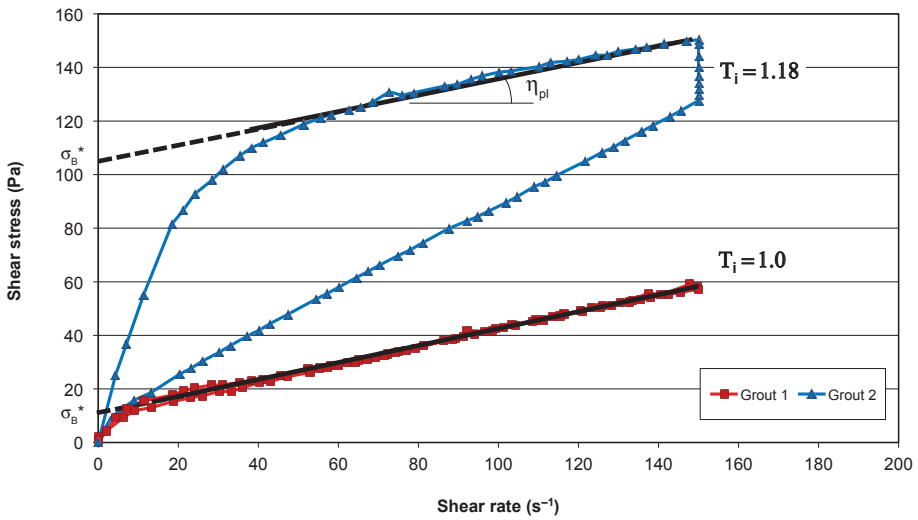



**Figure 3.3.3** Flow curves for two injection grouts: Plastic viscosity ($\eta_{pl}$) of grout 1 (red) and grout 2 (blue) are obtained from the slope of the linear portion of the curves as 0.33 Pa·s and 0.31 Pa·s, respectively. Apparent Bingham yield stress ($\sigma_B^*$) of grout 1 and grout 2 are 12.9 Pa and 105.6 Pa, and the thixotropy index (Ti) of grout 1 and grout 2 are 1.0 and 1.18, respectively. Grout 2 has a higher apparent Bingham yield stress, indicating that it will require higher applied pressure to begin flowing (i.e., back pressure in the syringe will be higher). Grout 2 also demonstrates thixotropy (i.e., when constant pressure is applied to a syringe, it will flow faster and faster). Both grouts have very similar plastic viscosities.

should be followed. Testing should be started within 4 minutes of mixing.
2. Attach the rotor and then the cup to the viscometer.
3. Remove the excess grout on the top of the rotor with a small spatula. Do not permit the grout to reach a level higher than the top of the rotor inside the cup.
4. Set the testing regime using the computer program:
   a. Stir at a shear rate of 0.02 $s^{-1}$ for 30 seconds until a stable shear stress reading is obtained.
   b. Increase the shear rate to 150 $s^{-1}$ within 120 seconds.
   c. Keep the shear rate at the maximum value for 60 seconds to determine the thixotropy.
   d. Decrease the shear rate to 0 within 120 seconds.
5. Turn on the external cooling system and start testing.

### Results

1. The plastic viscosity ($\eta_{pl}$) of the grout is calculated from the slope of the linear portion of the flow curve—shear stress ($\sigma$) in pascals as a function of shear rate ($\dot{\gamma}$) in $s^{-1}$, as shown in figure 3.3.3. The apparent Bingham yield stress ($\sigma_B^*$) in pascals is the shear stress value extrapolated for a shear rate of zero (i.e., the stress value where the extrapolated linear portion of the flow curve intersects the shear stress axis), as shown in figure 3.3.3.
2. The hysteresis between the flow curves obtained by the increasing (above curve) and decreasing shear rate characterizes thixotropy—i.e., a decrease in viscosity over time at a constant shear rate. A thixotropy index ($T_i$) is calculated as the ratio of the maximum and minimum shear stress values at 150 $s^{-1}$. If $T_i$ is greater than 1.0, the grout is thixotropic. The larger the $T_i$ value, the greater the thixotropic behavior.
3. The measurement is repeated on a fresh batch of grout. The average of two independent measurements is stated as the average plastic viscosity, average apparent yield stress, and average thixotropy index. The average value should not differ more than 5% from each measured value; otherwise, the test should be repeated.

## 3.4. Time of Setting by Vicat Needle

### Aim

This test is used to determine the time of initial and final setting of a hydraulic grout. The time of setting is of interest particularly in situations where a fairly rapid set may be desired (e.g., fragile plasters, vaults, large areas of loss) or cases in which it is necessary to know when supports can be removed. Desirable setting time should be determined on a case-by-case basis, depending on the type of voids or cracks to be grouted.

### Description

The method follows ASTM C 953—Standard Test Method for Time of Setting of Grouts for Preplaced-Aggregate Concrete in the Laboratory—and ASTM C 191—Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle. Grout is placed in a conical mold, and the time of setting is determined by periodic insertion of a Vicat needle until a predefined needle penetration depth is obtained. Setting is defined as the onset of rigidity and covers the transitional period when the grout changes its state from liquid to solid. The initial and final setting times are defined and measured as two arbitrary points in the development of rigidity. However, one may consider that the initial setting time represents approximately the time at which the grout reaches the limits of handling, and final setting time represents approximately the time at which hardening (i.e., the development of useful and measurable strength) begins. Even so, grout will lose its workability before initial set, and hardening will take place sometime after final set.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces

The rate of the hydration reaction in lime-based hydraulic injection grouts used for the conservation of architectural surfaces is much slower than the rate of the hydration reaction of grouts or mortars that include cement as a binder. As a result, testing with this method starts after 8 hours rather than after 3.00 ± 0.25 hours, as specified in ASTM C 953, or after 0.5 hour, as specified in ASTM C 191. This delay also makes the use of a manual Vicat needle apparatus challenging, since workdays need to be extended to accommodate testing between 8 to 12 hours. An automated Vicat apparatus, one that can be programmed to run without an operator present at all times, is recommended as a solution. When an automated Vicat apparatus is used and the specimen is left in the apparatus for a prolonged time (for example, up to 10 hours while measurements are taken every 15 minutes), the specimen should be placed in a container filled with saturated limewater to prevent drying.

### Notes

All materials are stored at the temperature (23°C ± 2°C) at which the mixing will be conducted and the test will be run. The temperature of the moist cabinet is also kept at 23°C ± 2°C. The RH of the laboratory is kept at 60% ± 10% during the experiment.

### Equipment and Materials

- automated Vicat apparatus (fig. 3.4.1) with 1 mm diameter needle and 300 g plunger weight
- glass baseplate (115 mm diameter)
- four conical plastic ring molds (60 mm top diameter, 70 mm bottom diameter, 40 mm high)
- grease (e.g., petroleum jelly, Vaseline, high-vacuum grease, etc.)
- hot glue gun and glue sticks
- trowel with a steel blade 100–150 mm in length, with straight edges
- crystallization dish or wide-mouth glass container (approximately 125 mm diameter and 60 mm high)
- saturated limewater enough to fill used crystallization dish or wide-mouth glass container when the specimen is in it
- thermometer accurate to within 1°C
- RH meter accurate to within 1%


**Figure 3.4.1** Automated Vicat apparatus (cement setting tester, appendix B).

### Specimen Preparation

1. Apply a thin film of grease to the base of the conical ring mold; place and center the greased conical ring on the glass baseplate.
2. Adhere the bottom of the conical ring mold to the glass baseplate with hot glue while applying pressure on the conical ring mold.
3. Within 2 minutes of mixing the grout, fill the ring mold slowly with grout until it is flush with the top edge.
4. Strike off the excess grout with a trowel held at a slight angle to the top of the mold. Take care not to compress the grout specimen.
5. Record the time of the completion of mold filling as the start of the setting time measurement ($t_0$).
6. Prepare at least four specimens and store the specimens in a moist cabinet at RH of 95% ± 5% and at 23°C ± 2°C.

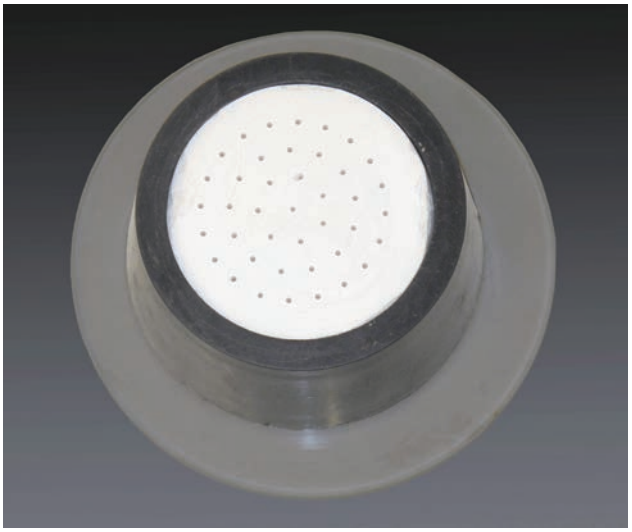


**Figure 3.4.2** Top surface of the tested grout specimen.

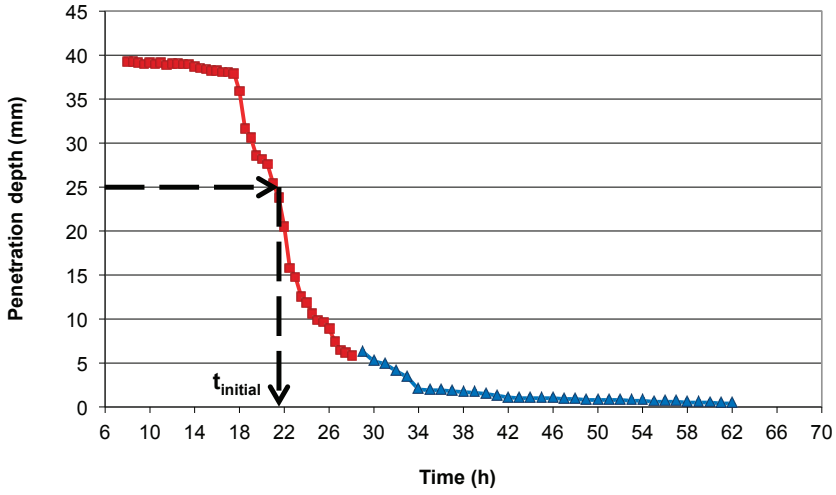
### Procedure

1. Remove one specimen from the moist cabinet after 8 hours; place it in a glass container. Position the container under the Vicat apparatus (fig. 3.4.1) and fill with saturated limewater.
2. Determine the penetration of the 1 mm needle at this time ($t_s$) and every 30 minutes ($\Delta t$) until a penetration of less than 25 mm is obtained (fig. 3.4.2). If a penetration less than 25 mm is not obtained, continue testing with the next specimen.
3. Once the penetration of less than 25 mm is obtained and all the possible penetration data from the specimen are collected, continue testing using the next specimen and determine the penetration depth every 60 minutes ($\Delta t$) until a penetration of less than 0.5 mm is obtained.
4. If a penetration less than 0.5 mm is not obtained after testing the fourth specimen, repeat the test and postpone the time of removal of the specimen from the moist cabinet, or program a delay for the start of the test in the machine using the previous test results.

### Results

1. Record the results of all penetration tests by plotting the depth of penetration as a function of time.
2. By interpolation, determine the time when a penetration of 25 mm is obtained. This is the initial setting time ($t_{initial}$) in hours (fig. 3.4.3).
3. The final setting time ($t_{final}$) in hours is the time when the needle does not visibly sink into the specimen—i.e., when the penetration depth is less than 0.5 mm. By interpolation, determine the time when a penetration depth of 0.5 mm is obtained (fig. 3.4.4).

**Figure 3.4.3** Penetration depth versus time curve for an injection grout, plotted using the results of two specimens. The results obtained from the first specimen are shown in red, and the results of the second specimen are shown in blue. The initial setting time of the grout is determined to be 21.5 hours.



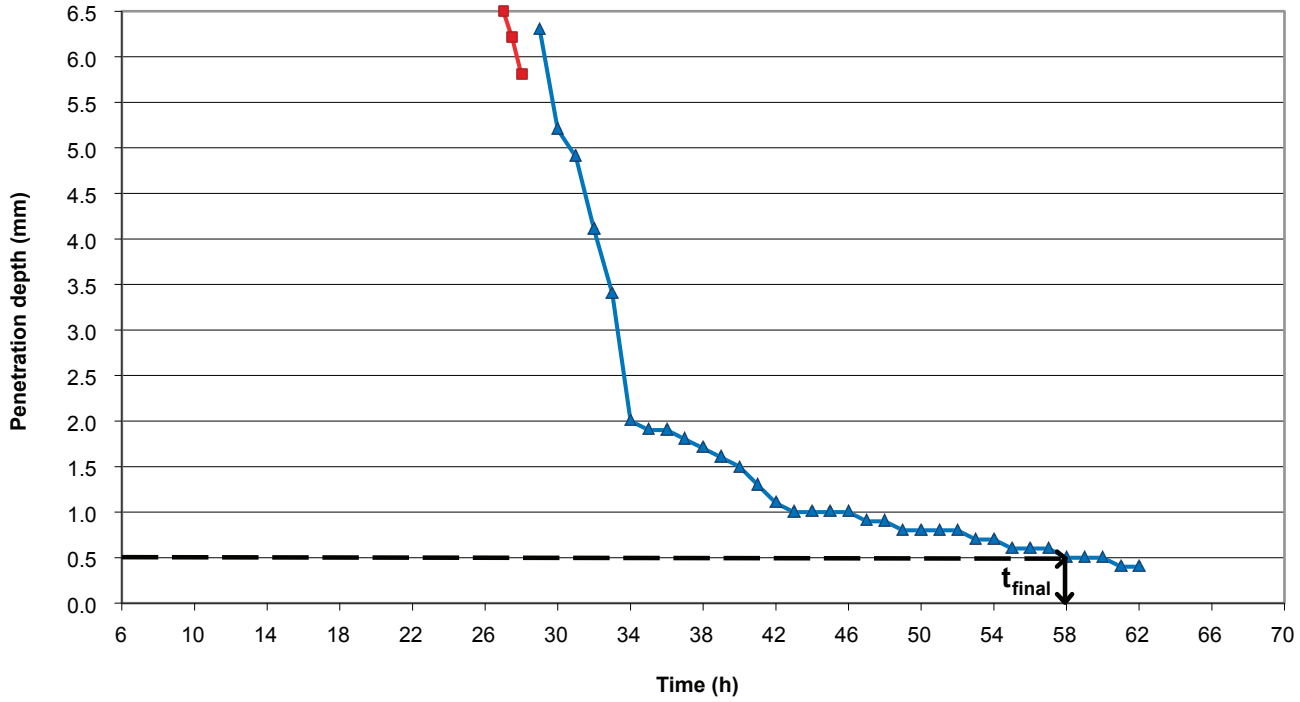



**Figure 3.4.4** Close-up graph of the penetration depth versus time curve of the injection grout shown in figure 3.4.3. The results obtained from the first specimen are plotted in red, and the results of the second specimen are plotted in blue. The final setting time of the grout is determined to be 58 hours.

4. The setting time is the average of two independent measurements when these measurements do not differ from each other by more than 30 minutes for initial setting time and 60 minutes for final setting time; otherwise, the test should be repeated.
5. An example data collection sheet is given in figure 3.4.5.

## TIME OF SETTING BY VICAT NEEDLE

| | | | | |
|---|---|---|---|---|
| **Grout name** | | **Room** | **Temperature (°C)** | |
| **Grout proportions** | | | **RH (%)** | |
| **Operator** | | **Date** | | |

**$t_0$:** Time when the specimen is prepared

**$t_s$:** Time when testing is started

**$\Delta t$ (min):** Time interval between two consecutive penetrations

| | Test 1 | | | | | Test 2 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | $t_0$ | | $t_s$ | | $\Delta t$ | $t_0$ | | $t_s$ | | $\Delta t$ |
| | Date | Hour:min | Date | Hour:min | Min | Date | Hour:min | Date | Hour:min | Min |
| 1 | | | | | | | | | | |
| 2 | | | | | | | | | | |
| 3 | | | | | | | | | | |
| 4 | | | | | | | | | | |

**$t_{inital}$ (h):** Time when the penetration of 25 mm is obtained

**$t_{final}$ (h):** Time when the penetration of 0.5 mm is obtained

| | Test 1 | | Test 2 | |
|---|---|---|---|---|
| | $t_{initial}$ (h) | $t_{final}$ (h) | $t_{initial}$ (h) | $t_{final}$ (h) |
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |

$t_{initial}$, average = _____ h

$t_{final}$, average = _____ h

**Figure 3.4.5** Data collection sheet for the time of setting by Vicat needle test method.

## 3.5. Shear Bond Strength

### Aim

This test is used to determine the shear bond strength between a grout and a stone substrate. It is important to note that it is not desirable for injection grouts used in the conservation of architectural surfaces to have bond strengths higher than the strength of the original material. In case of bond failure, the failure should be either at the interface between the substrate and grout, or in the grout and not in the original material. This test method is designed to limit the failure so that it occurs in the injection grout or at the bond interface (or in both places) by using substrates stiffer and stronger than both grout and bond. Therefore, it does not address the issues of failure occurring in the original material.

### Description

The sample configuration loosely follows ASTM D 905—Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading. The specimens are tested in compliance with EN 196-1: Methods of Testing Cement—Part 1: Determination of Strength. Two stone surfaces are adhered to each other using an injection grout, and after curing, the bond strength is determined in shear by compression loading. This test method is intended to be used for comparing the bonding abilities of injection grouts, but it cannot be assumed that the method measures the true shear bond strength or that the results can be taken as a design value of shear bond strength for use in the field. Many factors, including the properties of the substrate and grout, the size of the specimens, and the testing fixtures and conditions, can affect the bond strength. This procedure aims to minimize the possible variations of the substrate (e.g., type of substrate, surface texture, and water absorption capacity) and testing procedure, in order to allow comparison of the bonding abilities of different injection grouts.

Substrate properties are controlled by preparing all specimens from stone of the same type and source. Bluestone sandstone from Pennsylvania was used for the development of this test procedure at the GCI. All the stone samples were flat-sawn. The capillary water absorption coefficients (A) of all stone samples (using the surface that would be used as the bonding surface) were measured, and those having A of $1.0 \pm 0.2$ g·m$^{-2}$·s$^{-1/2}$ were selected for use. Since water absorption is minimal, prewetting is omitted. A detailed test procedure for measuring the capillary water absorption coefficient is given in Laboratory Testing Procedures 2.7. If the substrate is to be prewetted prior to testing, capillary water absorption measurements can be omitted.

### Equipment and Materials

- stone cut into three different sections (fig. 3.5.1); six pieces are needed for each section type: (A) 77.0 mm × 38.5 mm × 19 mm; (B) 77.0 mm × 38.5 mm × 9.5 mm; and (C) (spacer): 77.0 mm × 11.5 mm × 11.5 mm
- two 14 gauge stainless steel blunt cannulae (dispensing needles) (outer diameter, 2.1 mm; inner diameter, 1.70 mm; length, 25.4 mm) as spacers for 2.1 mm spacing (fig. 3.5.2)
- clear (stationery) adhesive tape
- aluminum foil waterproofing duct tape for general use (approximately 100 mm wide)
- 20 mL syringe
- 4 mil plastic sheet (e.g., window insulation sheet)
- Instron universal mechanical testing machine or any mechanical testing instrument equipped with compression fixtures and a sample holder
- oven

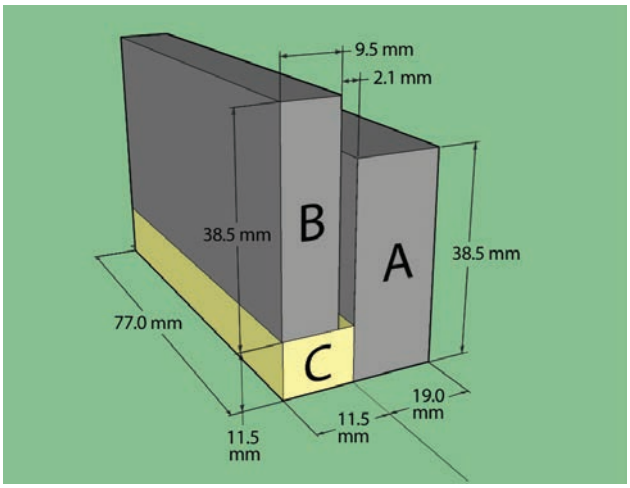



**Figure 3.5.1** Specimen assembly.

**Figure 3.5.2** Preparation of specimen assembly.

### Specimen Preparation

1. Oven-dry all the stone substrates needed for preparing six specimens: dry at 105°C ± 1°C for 2 days, then cool to 23°C ± 2°C.
2. Tape all surfaces of the stone spacer (C) using clear adhesive tape to prevent grout adherence.
3. Position three stone samples (A, B, and C) as shown in figure 3.5.1.
4. Insert two cannulae as spacers between the vertical stone substrates (A and B), and tape around them with aluminum foil duct tape (fig. 3.5.2).
5. Complete the substrate assembly preparation by taping the bottom (B and C) with aluminum foil tape and removing the two cannulae.
6. Slowly inject the grout into the crevice, being careful not to entrap any air bubbles (fig. 3.5.3).
7. To remove any entrapped air, tap the substrate assembly 10 times by slowly hitting it on the counter.
8. Store the specimens in a moist cabinet at RH of 95% ± 5% and at 23°C ± 2°C until testing.
9. Specimens are tested after at least 2 months or preferably after 6 months of curing.

### Prewetting

The substrate assembly can be prewetted by filling the crevice between the vertical stone pieces A and B with water (fig. 3.5.3) before the grout is injected. Allow the stone to absorb the water for 5 minutes, empty the excess water, and drain for another 5 minutes. Follow the specimen preparation steps from 6 through 9. The condition of the stone (oven dried or prewetted) should be noted.

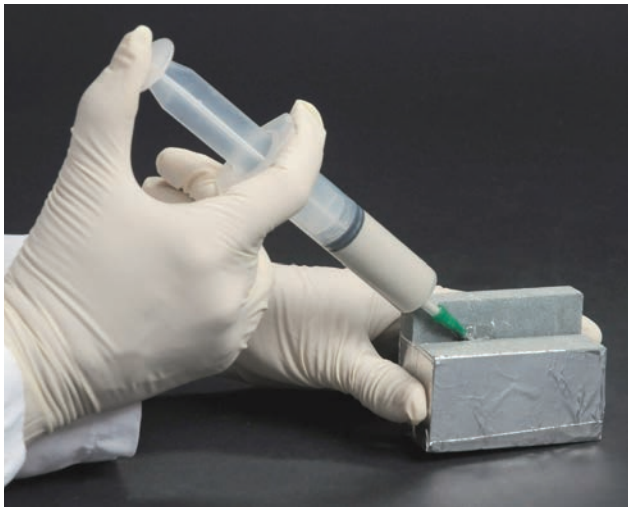


**Figure 3.5.3** Injecting grout into the crevice between the stone surfaces.

### Procedure

1. Test the specimens immediately after removing them from the moist cabinet. During testing, cover the untested specimens with a plastic sheet to avoid drying.
2. Remove all the aluminum tape and the stone spacer just before placing the specimen into the machine.
3. The specimens are held in place during testing, as shown in figure 3.5.4a, to prevent any eccentric loading. The specimen is set 6 mm deep into the holder and is held in place by screws inserted from the sides of the holder, as seen in figure 3.5.4b.
4. Test the specimens under a loading rate increase of 2400 ± 200 $N \cdot s^{-1}$ in compression (figs. 3.5.5a and 3.5.5b).
5. Measure the length (l) and width (w), both in millimeters, of the grouted area shown in red on the tested specimens in figure 3.5.6.

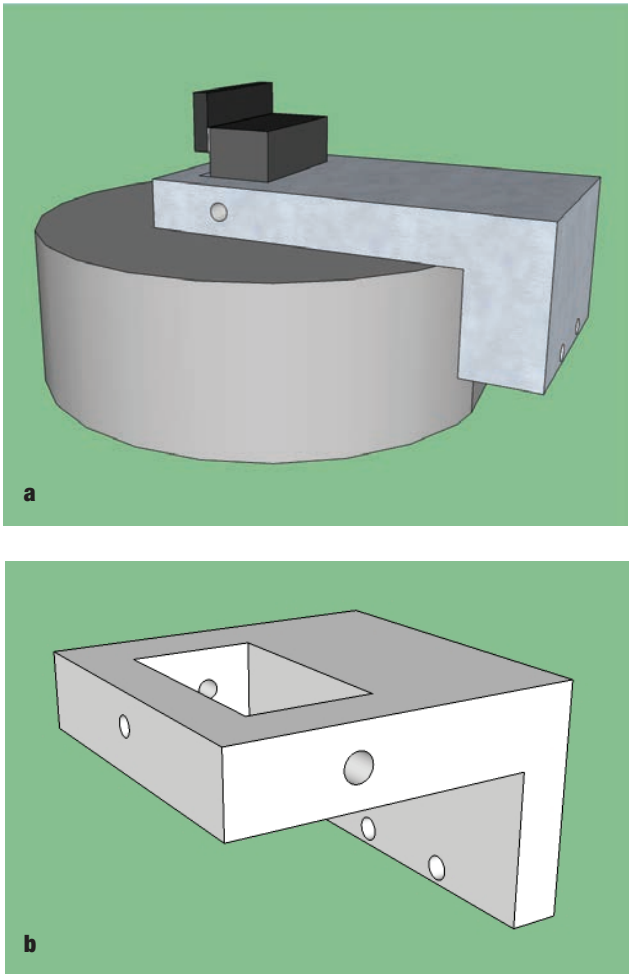
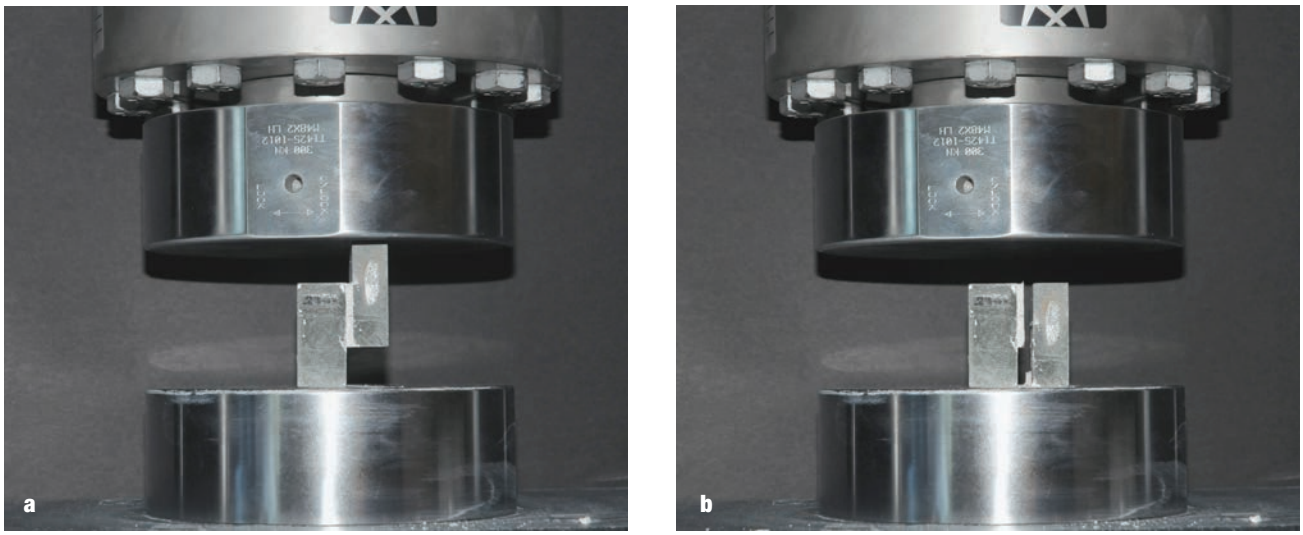



**Figure 3.5.4a and 3.5.4b** Specimen, holder, and base plate (a), and detail of holder (b) (not to scale).




**Figures 3.5.5a and 3.5.5b** Before testing (a) and after testing (b); for clarity, the specimen holder is not pictured.

## Results

1. Record the maximum load during the test as the breaking load, F, in newtons, and calculate the shear bond strength, $f_{sb}$, in $N \cdot mm^{-2}$ using the following relationship:

$$f_{sb} = \frac{F}{w \times l} \quad (3.5.1)$$

where $w$ is the width, and $l$ is the length of the grouted area (fig. 3.5.6), both in millimeters.

2. Calculate the average shear bond strength using the results of six specimens obtained for each grout. Discard any individual result if the average and the independent result differ more than 20%. Average shear bond strength should be obtained by at least three individual results; otherwise, the test should be repeated with another six specimens.
3. An example data collection sheet for this procedure is given in figure 3.5.7.

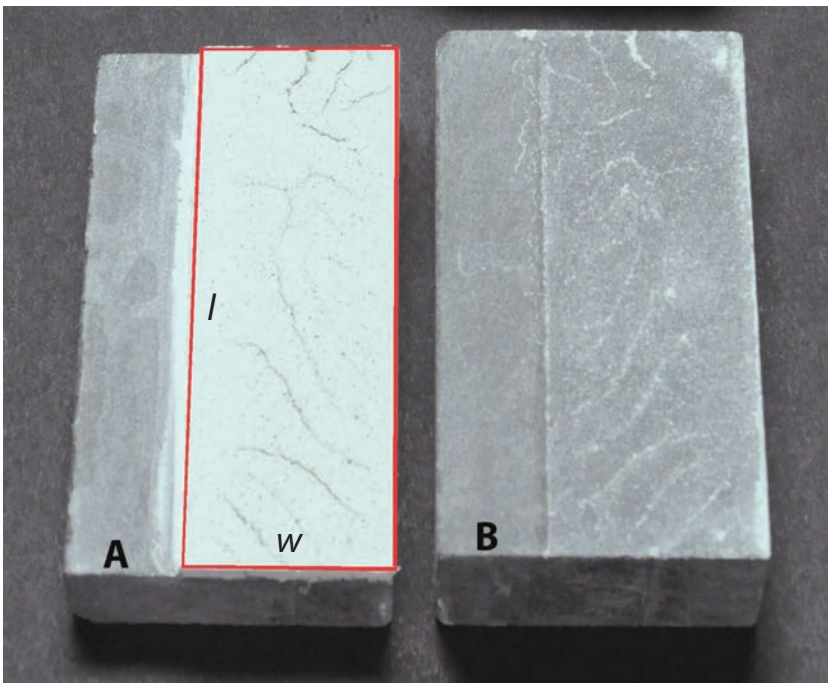



**Figure 3.5.6** Tested specimen. Related stone substrate pieces (A and B) are shown. The outlined area on stone substrate A indicates the effective grouting area (i.e., multiplication of length, *l*, and width, *w*, which is used for calculating the shear bond strength).

## SHEAR BOND STRENGTH

| | | | |
|---|---|---|---|
| **Grout name** | | **Compressive loading rate** | $2400 \pm 200\ N \cdot s^{-1}$ |
| **Grout proportions** | | **Age of specimen (days)** | |
| **Operator** | | **Date** | |

**w (mm):** Width of failed grout area
**l (mm):** Length of failed grout area
**F (N):** Breaking load
**$f_{sb}$ ($N \cdot mm^{-2}$):** Shear bond strength

**EQUATION**

$$f_{sb} = \frac{F}{w \times l}$$

| Specimen no. | w (mm) | l (mm) | F (N) | $f_{sb}$ ($N \cdot mm^{-2}$) |
|---|---|---|---|---|
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |

**$f_{sb}$, average =** _____ **$N \cdot mm^{-2}$**

**Figure 3.5.7** Data collection sheet for the shear bond strength test method.

PART II

# Field Testing Procedures

# 4. Field Testing Procedures



## 4.1. Injectability with Syringe

### Aim

The aim of this field test is to compare the ability of a grout to fill a prewetted or dry capillary network of different granular materials under pressure. Injectability is a critical property for injection grouts, which must be suitable for injection through a syringe or tubing to fill internal cracks and voids.

### Description

Grout is poured into a vertically held syringe that is partially filled with granular material, and pressure is applied on the grout using the syringe plunger. This field test can also be used for testing nonhydraulic injection grouts.

### Equipment and Materials

- crushed travertine or crushed brick (particle size, 2–4 mm); alternatively, debris removed from the location where the injection grouting will be conducted can be used
- 100 mL distilled water
- two 60 mL syringes
- 20 mL syringe
- ruler in millimeters

### Procedure

1. Pour 20 mL granular material into an empty 60 mL syringe with the plunger removed (fig. 4.1.1).
2. Hold the syringe in a vertical position and tap the side of it 10 times with a finger while slowly rotating the syringe.
3. Inject 20 mL grout from the 20 mL syringe into a 60 mL syringe (fig. 4.1.2).
4. Insert the plunger into the syringe and apply gentle pressure to the plunger (fig. 4.1.3).
5. Repeat steps 1–3 using the second 60 mL syringe.
6. If damp medium is used, pour 100 mL of distilled water into the syringe partially filled with granular material and let the excess water drain for 5 minutes.

**Figure 4.1.1** Pouring 20 mL crushed brick into a 60 mL syringe.

### Results

1. Classify the injectability of the grout as: easy (E)—if grout flows out of the syringe tip when pressure is applied (fig. 4.1.4); feasible (F)—if grout reaches the tip of the syringe but does not flow through when pressure is applied; or difficult ($D_L$)—if grout stops before reaching the tip of the syringe when pressure

**Figure 4.1.2** Injecting 20 mL grout into a 60 mL syringe.

**Figure 4.1.3** Inserting the plunger into the syringe.

**Figure 4.1.4** Applying pressure to the plunger either until grout comes out the tip or until grout stops moving through the granular material.

is applied. Using the millimeter ruler, record the distance reached by the grout (L) in millimeters.

2. Repeat the test for the same material type until two experimental runs providing the same classification are obtained. For the grouts classified as difficult, the average level reached by the grout is calculated from the distances reached by two independent runs. The average distance reached in the syringe of the two runs should not differ more than 25% from the individual results; otherwise, the test should be repeated.
3. Record results on a data collection sheet; see figure 4.1.5 for an example.

## INJECTABILITY WITH SYRINGE

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |

**EASY (E):** If grout flows out of the syringe tip when pressure is applied

**FEASIBLE (F):** If grout reaches the tip of the syringe but does not flow through when pressure is applied

**DIFFICULT ($D_L$):** If grout halts before reaching the tip of the syringe when pressure is applied; record the distance reached (L) in mm

| | Travertine-wet | | Travertine-dry | | Brick-wet | | Brick-dry | |
|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| **Classification** | | | | | | | | |

**Figure 4.1.5** Data collection sheet for the injectability with syringe test method.

## 4.2. Flow with Syringe

### Aim

The aim of this field test is to compare the ability of a grout to pass through a prewetted or dry capillary network of different granular materials under gravitational force.

### Description

Grout is poured into a vertically held syringe (without a plunger) that is partially filled with granular material, and the penetration of grout into the granular material in the syringe is observed. This field test can also be used for testing nonhydraulic injection grouts.

### Equipment and Materials

- crushed travertine or crushed brick (particle size, 2–4 mm); alternatively, debris removed from the location where the injection grouting will be conducted can be used
- 100 mL distilled water
- two 60 mL syringes
- 20 mL syringe
- clear rigid plastic tube or graduated cylinder with approximately 45 mm diameter and longer than 15 cm
- ruler in millimeters
- wristwatch

### Procedure

1. Pour 20 mL granular material into an empty 60 mL syringe with the plunger removed (fig. 4.2.1).
2. Hold the syringe in a vertical position and tap the side of it 10 times with a finger while slowly rotating the syringe.
3. Inject 20 mL grout from the 20 mL syringe into a 60 mL syringe (fig. 4.2.2) while holding the 60 mL syringe in a vertical position or while supporting the syringe by placing it in a clear rigid plastic tube or graduated cylinder standing upright.
4. Start measuring the time.
5. Repeat steps 1 and 2 using the second 60 mL syringe.
6. If damp medium is used, pour 100 mL of distilled water into the syringe partially filled with granular material and let the excess water drain for 5 minutes.

**Figure 4.2.1** Pouring 20 mL crushed brick into a 60 mL syringe.

**Figure 4.2.2** Injecting 20 mL grout into a 60 mL syringe. Note that the syringe with grout and granular material is held vertically and supported by a rigid plastic tube.

### Results

1. Classify the flow of the grout as: easy (E)—if grout flows through the tip of the syringe in 5 minutes or less; feasible (F)—if grout reaches the tip of the syringe but does not flow through it in 5 minutes; difficult ($D_L$)—if grout halts before reaching the tip of the syringe in 5 minutes (fig. 4.2.3). Record the distance reached by the grout (L) in millimeters.
2. Repeat the test for the same medium until two experimental runs providing the same classification are obtained. For the grouts classified as difficult, the average level reached by the grout is calculated from the levels reached by two independent runs. The average level reached in the syringe of the two runs should not differ more than 25% from the individual results; otherwise, the test should be repeated.
3. Record results on a data collection sheet; see figure 4.2.4 for an example.

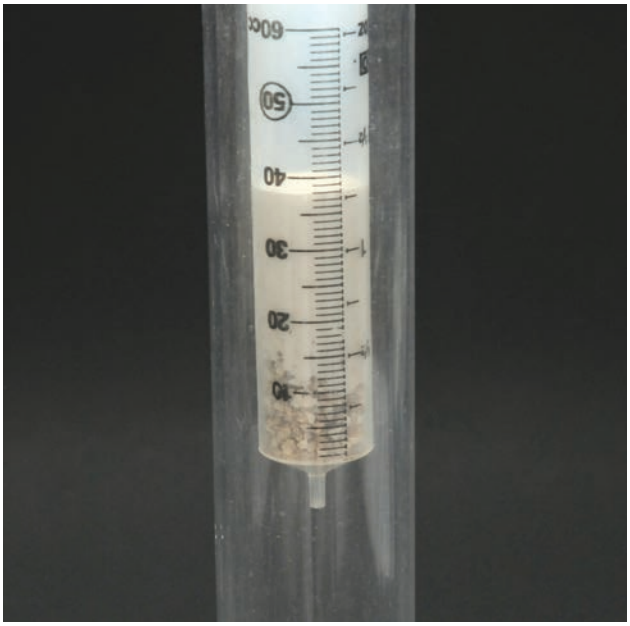



**Figure 4.2.3** Grout penetrating into crushed travertine. No grout has flowed out within 5 minutes; therefore, this grout is classified as difficult.

## FLOW WITH SYRINGE

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |

**EASY (E):** If grout flows through the tip of the syringe in 5 minutes

**FEASIBLE (F):** If grout reaches the tip of the syringe but does not flow through it in 5 minutes

**DIFFICULT ($D_L$):** If grout halts before reaching the tip of the syringe in 5 minutes; record the distance reached (L) in mm

| | Travertine-wet | | Travertine-dry | | Brick-wet | | Brick-dry | |
|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| **Classification** | | | | | | | | |

**Figure 4.2.4** Data collection sheet for the flow with syringe test method.

## 4.3. Flow on Plastered Tile

### Aim

The aim of this field test is to compare the ability of different grouts to flow within a vertical crevice in plaster. The distance a specific grout flows is an indication of how far it can flow through a network of cracks or voids. This test can be used in the field as a simple way of comparing the flow behavior of different grouts to determine which grout to use on a case-by-case basis.

### Description

A fixed volume of grout is poured into a vertical crevice created in a coarse plastered tile, and the distance it flows is determined. This test can also be used for testing nonhydraulic injection grouts.

### Notes

Mortar used for repairs on site can be used to plaster the reverse of the tile for this test. The following mortar proportions and ingredients, given here as a reference, were used at the GCI to prepare the plaster.

### Equipment and Materials

- standard sand (EN 196-1)
- superventilated pozzolana
- lime putty
- mixing bowl or large plastic container to mix the plaster
- mortar mixer if available
- trowel
- glazed or unglazed ceramic tile (33 × 25 × about 1 cm thick)
- spray bottle
- distilled water
- pointed cutting tool for creating flow paths (e.g., wood tool for ceramics)
- ruler in millimeters
- 10 mL syringe
- thermometer
- humidity gauge
- plastic bag or moist cabinet larger than the size of the ceramic tile

### Preparation of Plastered Tile

1. Pour 2.5 parts (by volume) standard sand and 0.5 parts (by volume) superventilated pozzolana into a container and mix with mortar mixer or trowel.
2. Add 1 part (by volume) lime putty and mix thoroughly with the mortar mixer at low speed, or mix manually with a trowel.
3. Using the spray bottle, prewet the reverse of the tile with water.
4. Spread plaster over the reverse of the tile to a thickness of 3–4 cm.
5. Smooth the surface with the trowel.
6. While the plaster is still wet, use the ruler and a cutting tool to make parallel V-shaped channels approximately 0.5 cm wide and 2–3 cm apart (fig. 4.3.1).

**Figure 4.3.1** Making parallel V-shaped channels using a ruler and cutting tool.

**Figure 4.3.2** Prepared flow tile after setting.

**Figure 4.3.3** Injecting 10 mL grout into a channel.

7. Store the plastered tile in a tightly closed plastic bag (or in a moist cabinet at RH greater than 95%) at 20°C ± 5°C for 2 weeks.
8. At the end of 2 weeks, remove the plastered tile from the plastic bag (or from the moist cabinet) and keep it near the location where the grouting will be executed for at least 2 more weeks and until it is strong enough to be placed on its edge. Record the maximum and minimum RH and temperature weekly.

### Procedure

1. Set the plastered tile on its edge so that the channels are vertical (fig. 4.3.2).
2. Record the temperature and humidity.
3. Using the 10 mL syringe, inject 10 mL grout into the top of a channel, as shown in figure 4.3.3.

### Results

1. Once the grout has stopped flowing, use a ruler to measure the distance (L) the grout has flowed down the channel in millimeters. The flow of the grout can also be classified according to the measured distance: low (L)—if grout flows a distance less than 100 mm; medium (M)—if grout flows a distance less than 200 mm; high (H)—if grout flows a distance more than 200 mm. Note if grout is collected at the bottom of the channel (fig. 4.3.4). The farther down the channel the grout travels, the better the flow.
2. Repeat the test until two experimental runs providing the same classification are obtained. The average of two independent measurements is the average flow distance. Average distance should not differ more than 25% from each measured value; otherwise, the test should be repeated.
3. Record results on a data collection sheet; see figure 4.3.5 for an example.

**Figure 4.3.4** The distance the grout has flowed is recorded. In this case, grout demonstrated high flow and accumulated at the bottom of the tile.

## FLOW ON PLASTERED TILE

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |

| | Week no. | Temperature (°C) | | RH (%) | |
|---|---|---|---|---|---|
| | | Max | Min | Max | Min |
| Plaster | 1 | | | | |
| | 2 | | | | |

**L (mm):** Distance traveled by grout

**LOW (L):** If grout flows a distance less than 100 mm

**MEDIUM (M):** If grout flows a distance less than 200 mm

**HIGH (H):** If grout flows a distance more than 200 mm. Note if grout is collected at the bottom of the channel.

| Specimen no. | Temperature (°C) | RH (%) | L (mm) | Classification |
|---|---|---|---|---|
| 1 | | | | |
| 2 | | | | |

**L, average =** _____ mm

**Classification** _____

**Figure 4.3.5** Data collection sheet for the flow on plastered tile test method.

## 4.4. Expansion and Bleeding

### Aim

This test is used in the field to determine the amount of expansion and accumulation of bleed water that forms over time at the surface of freshly mixed grout. Grouts that are well formulated and properly proportioned should not segregate or bleed visibly. Excessive segregation or bleeding of a grout will change its properties and cause clogging during injection. A suggested final bleeding should be less than 0.4%.

### Description

This test follows ASTM C 940—Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory—and the laboratory procedure for the expansion and bleeding of grouts (section 2.2 in this volume), with the exception of sample volume, which has been modified for field use. Grout is placed in a graduated cylinder, and the change in total volume and the rate of accumulation of bleed water on the surface of the grout are measured over a period of time. The volume of bleed water with respect to the total volume of grout is an indication of the separation of the liquid and solid phases. It is important to determine the expansion of a lime-based hydraulic grout when the injection grout includes additives that facilitate expansion. This test can also be used for testing nonhydraulic injection grouts.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces in the Field

The volume of grout used is reduced from 800 ± 10 mL, as specified in ASTM C 940, to 80 ± 10 mL. All test equipment and materials are stored in closed containers in the location where the test will be run.

### Equipment and Materials

- 100 mL plastic or glass graduated cylinder reading to 1 mL
- 25 mL plastic or glass graduated cylinder reading to 1 mL
- 10 mL syringe with cannula (#20 or higher)
- stopwatch
- thermometer
- Parafilm or thin plastic wrap (e.g., food wrap)


**Figure 4.4.1** Grout in a graduated cylinder.


**Figure 4.4.2** Bleed water at the top of the 100 mL graduated cylinder.

### Procedure

1. Place the 100 mL graduated cylinder on a level surface free of vibration.
2. Mix the grout and record the ambient temperature.
3. Pour the grout into the graduated cylinder until the volume of the sample is 80 ± 10 mL (fig. 4.4.1). Volume measurements should begin within 3 minutes after the grout is mixed.
4. Cover the top with Parafilm or plastic wrap to prevent evaporation of the bleed water.
5. Record the total initial volume of the sample ($V_0$).
6. Record the volume at the upper surface of the bleed water layer ($V_t$) and at the upper surface of the grout ($V_g$), to the nearest 1 mL at 15 minute intervals for the first 60 minutes; thereafter, do this at hourly intervals until two successive readings show no further change in volume of the grout.
7. At the end of the test, transfer the bleed water (fig. 4.4.2) into a 25 mL graduated cylinder by tilting the cylinder and drawing the water off with a

syringe. Record the final volume of the bleed water ($V_w$) to the nearest 0.5 mL.

### Results

1. Calculate the expansion of the grout (E) to the nearest 1% for each prescribed interval:

$$E = \frac{V_g - V_0}{V_0} \times 100 \qquad (4.4.1)$$

where $V_0$ is the volume of the sample at the beginning of the test, and $V_g$ is the volume of the grout portion of the sample at prescribed intervals, measured at the upper surface of the grout, both in milliliters.

2. Calculate the combined expansion of the grout (CE) to the nearest 1% for each prescribed interval:

$$CE = \frac{V_t - V_0}{V_0} \times 100 \qquad (4.4.2)$$

where $V_0$ is the volume of the sample at the beginning of the test, and $V_t$ is the volume of the sample at prescribed intervals, measured at the upper surface of the water layer, both in milliliters.

3. Calculate the bleeding of the grout (B) to the nearest 1% for each prescribed interval:

$$B = \frac{V_t - V_g}{V_0} \times 100 \qquad (4.4.3)$$

where $V_0$ is the volume of the sample at the beginning of the test; $V_t$ is the volume of the sample at prescribed intervals, measured at the upper surface of the water layer; and $V_g$ is the volume of the grout portion of the sample at prescribed intervals, measured at the upper surface of the grout, all in milliliters.

4. Calculate the final bleed water (FB) as a percentage of the initial volume of the grout, to the nearest 1%:

$$FB = \frac{V_w}{V_0} \times 100 \qquad (4.4.4)$$

where $V_0$ is the volume of the sample at the beginning of the test, and $V_w$ is the volume of decanted bleed water, both in milliliters.

5. The average of two independent measurements is stated as E, CE, and B at each interval, and the average of two independent measurements is stated as FB. The average value should not differ more than 10% from each measured value; otherwise, the test should be repeated.

6. An example data collection sheet for this procedure is given in figure 4.4.3.

## EXPANSION AND BLEEDING

| | | | |
|---|---|---|---|
| **Grout name** | | **Ambient temperature (°C)** | |
| **Grout proportions** | | | |
| **Operator** | | **Date** | |

**$V_0$ (mL):** Volume of the sample at the beginning of the test

**$V_t$ (mL):** Volume of the sample at prescribed intervals, measured at the upper surface of the water layer

**$V_g$ (mL):** Volume of grout portion of sample at prescribed intervals, measured at the upper surface of grout

**$V_w$ (mL):** Volume of decanted bleed water

## EQUATIONS

| | |
|---|---|
| Expansion, E (%) = $\frac{V_g - V_0}{V_0} \times 100$ | Combined expansion, CE (%) = $\frac{V_t - V_0}{V_0} \times 100$ |
| Bleeding, B (%) = $\frac{V_t - V_g}{V_0} \times 100$ | Final bleeding, FB (%) = $\frac{V_w}{V_0} \times 100$ |

| | Test 1 | | | | | Test 2 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Hour:min** | **$V_t$ (mL)** | **$V_g$ (mL)** | **E (%)** | **CE (%)** | **B (%)** | **$V_t$ (mL)** | **$V_g$ (mL)** | **E (%)** | **CE (%)** | **B (%)** |
| **0:15** | | | | | | | | | | |
| **0:30** | | | | | | | | | | |
| **0:45** | | | | | | | | | | |
| **1:00** | | | | | | | | | | |
| **2:00** | | | | | | | | | | |
| **3:00** | | | | | | | | | | |
| **4:00** | | | | | | | | | | |
| **5:00** | | | | | | | | | | |
| **6:00** | | | | | | | | | | |
| **7:00** | | | | | | | | | | |
| **8:00** | | | | | | | | | | |

| | Test 1 | Test 2 |
|---|---|---|
| **$V_0$ (mL)** | | |
| **$V_w$ (mL)** | | |
| **FB (%)** | | |

**Figure 4.4.3** Data collection sheet for the expansion and bleeding test method.

## 4.5. Wet Density

### Aim

This test is used in the field to determine the wet density of a grout. Wet density becomes an important parameter if added weight as a result of grouting might cause structural instability or failure of the architectural surfaces. Examples of this would be the grouting of large voids behind architectural surfaces on ceilings and vaults.

### Description

This test follows the procedure of ASTM C 185—Standard Test Method for Air Content of Hydraulic Cement Mortar (section 9.4)—and the laboratory procedure for wet density (section 2.3 in this volume), with the exception of sample volume, which has been modified for field use. A syringe is filled with grout and weighed. The volume of the syringe and the measured weight of the grout are used to calculate the wet density. This test can also be used for testing nonhydraulic injection grouts.

### Equipment and Materials

- two 5 mL syringes
- balance accurate to within 0.1 g
- paper towel(s)

### Procedure

1. Tare the balance with an empty 5 mL syringe.
2. Pull 5 mL grout into the syringe and remove air bubbles by tapping with finger (fig. 4.5.1) until all air bubbles are removed.
3. Clean the tip of the syringe and wipe off all grout and water adhering to the outside of the syringe.
4. Weigh the filled syringe ($M_g$) (fig. 4.5.2).

**Figure 4.5.1** A syringe filled with grout is tapped to remove air bubbles.

### Results

1. Calculate the wet density of the grout ($\rho_{wet}$) in grams per cubic centimeter ($g \cdot cm^{-3}$) using the following formula:

$$\rho_{wet} = \frac{M_g}{5} \qquad (4.5.1)$$

   where $M_g$ is the weight of the grout in grams.
2. The average of two independent measurements is stated as the average wet density. The average value should not differ more than 10% from each measured value; otherwise, the test should be repeated.
3. An example data collection sheet for this procedure is given in figure 4.5.3.


**Figure 4.5.2** A grout-filled syringe is weighed.

## WET DENSITY

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |

**$M_g$ (g):** Weight of the grout in syringe

**$\rho_{wet}$ ($g \cdot cm^{-3}$):** Wet density of the grout

### EQUATION

$$\rho_{wet} = \frac{M_g}{5}$$

| Specimen no. | $M_g$ | $\rho_{wet}$ ($g \cdot cm^{-3}$) |
|---|---|---|
| 1 | | |
| 2 | | |

$\rho_{wet}$, average = _____ $g \cdot cm^{-3}$

**Figure 4.5.3** Data collection sheet for the wet density test method.

## 4.6. Drying Shrinkage

### Aim

This test is used in the field to determine the change of volume of a grout after drying. Volumetric stability of an injection grout is an important parameter directly affecting adhesion and durability. This test also provides information on the water content of the grout leading to volumetric instability. While increased water content may appear to improve some properties, such as injectability, it will decrease volumetric stability and lead to the formation of cracks, which in turn will cause loss of the bond between the grout and the substrate layers and loss of grout strength.

### Description

A container is filled with grout, and the dimensional changes, including cracking, of the grout are observed as the grout cures. During the testing, the grout-filled containers are placed near the location where the grouting will be conducted. This test can also be used for testing nonhydraulic injection grouts.

### Notes

The experiment can be run by injecting grout directly into weighing boats or similar shallow trays, or by injecting grout into prepared "mortar cups" (described below). The second setup aims to address the effect of absorption by the substrate on early drying shrinkage, in addition to the effect of evaporation on shrinkage, which is not taken into account if a nonporous setup (weighing boat) is used. The following procedure explains how to determine and classify shrinkage by the use of mortar cups. Mortar used for repairs on site can be employed to make mortar cups. The following mortar proportions and ingredients were used at the GCI to prepare the mortar cups and are given here as a reference.

### Equipment and Materials

- standard sand (EN 196-1)
- superventilated pozzolana
- lime putty
- mixing bowl or large plastic container for mixing mortar
- mortar mixer or trowel
- spatula
- plastic tube approximately 50 mm in diameter
- two weighing boats or similar shallow 30–50 mm deep containers at least 20 mm larger in diameter than the plastic tube described above
- 20 mL syringe
- thermometer
- humidity gauge
- caliper, ruler in millimeters, or a crack comparator card
- wristwatch
- plastic bags or moist cabinet

### Preparation of Mortar Cups

Prepare a minimum of two mortar cups per grout as follows:

1. Pour 2.5 parts (by volume) standard sand and 0.5 parts (by volume) superventilated pozzolana into a container and mix with mortar mixer or trowel.
2. Add 1 part (by volume) lime putty and mix thoroughly with a mortar mixer at low speed, or mix manually with a trowel.
3. Pour the mortar into a small plastic weighing boat or container, fill approximately 30 mm deep, and smooth the surface with a spatula (fig. 4.6.1).
4. Press one end of the plastic tube halfway (approx. 15 mm deep) into the mortar (not all the way to the bottom), and rotate it to hollow out a cylindrical cavity in the mortar (fig. 4.6.2).
5. Remove the plastic tube and remove excess mortar from inside the cylindrical cavity with the spatula to create a flat bottom (fig. 4.6.3). Reuse the plastic tube to prepare as many mortar cups as needed.

**Figure 4.6.1** A weighing boat or shallow container filled with mortar.

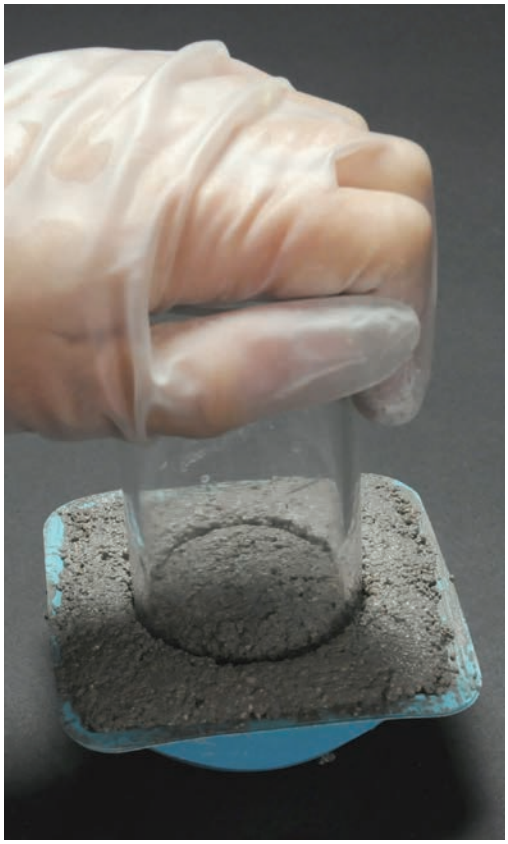


**Figure 4.6.2** Plastic tube being pressed and rotated to approximately half the depth of the mortar in the cup.

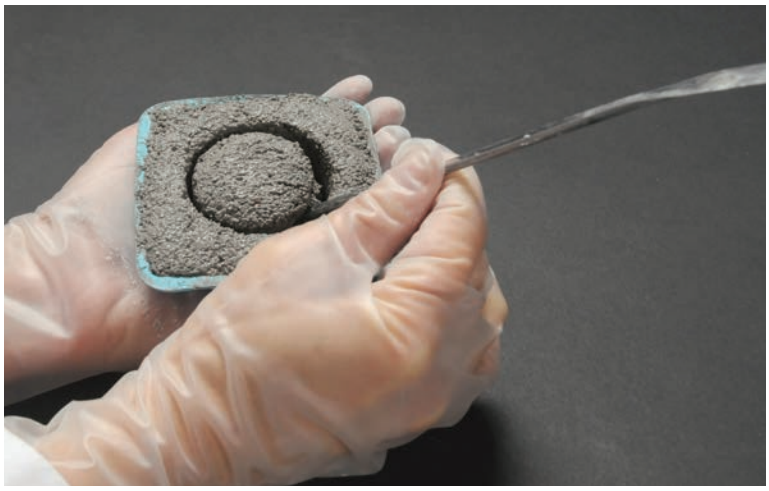


**Figure 4.6.3** Removing excess mortar from the cavity.

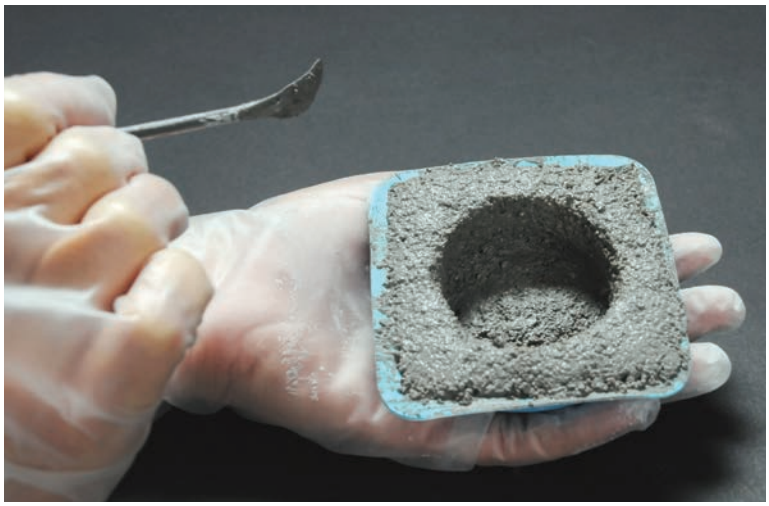


**Figure 4.6.4** Fresh mortar cup.

6. Store the mortar cups (fig. 4.6.4) in tightly closed plastic bags (or in the moist cabinet at RH greater than 95%) at 20°C ± 10°C for 2 weeks.
7. At the end of 2 weeks, remove the mortar cups from plastic bags (or from the moist cabinet) and keep them near the location where the grouting will be conducted for at least 2 more weeks.

### Procedure

1. Using a syringe without a cannula, inject 20 mL grout into shallow plastic container or mortar cup cavity (fig. 4.6.5).
2. Tap the container 10 times to spread the grout, and remove any entrapped air by slowly hitting the container on the counter.
3. Prepare a second specimen following steps 1 and 2; a total of two specimens per grout are needed.

**Figure 4.6.5** Grout being injected into a mortar cup.

4. Record the time and date of preparation.
5. Leave the grout-filled plastic containers or mortar cups in the area where the grouting will take place. Record the RH and ambient temperature.
6. Wait at least 24 hours.
7. Measure the gap between the grout sample and the sides of the plastic container or cylindrical mortar cup cavity. Record the maximum measured gap.
8. If any cracking has occurred in the grout itself, measure the maximum crack size.
9. Record the time and date of measurements and the ambient temperature and RH. It is recommended that the process of obtaining measurements be continued until two successive measurements are the same.

### Results

1. Classify the shrinkage of the grout as: no drying shrinkage (NS)—no visible separation between grout and mortar and no visible cracking in the grout (fig. 4.6.6); medium drying shrinkage (MS)—a separation of less than 0.5 mm between grout and mortar and/or a maximum crack size of less than 0.5 mm in the grout (fig. 4.6.7); or high drying shrinkage (HS)—a separation of more than 0.5 mm between grout and mortar and/or a maximum crack size of more than 0.5 mm in the grout (fig. 4.6.8).
2. Report the drying shrinkage classification when both specimens provide the same classification; otherwise, the test should be repeated with two other specimens.
3. Report all temperature and humidity measurements.
4. Record measurements and results on a data collection sheet; see figure 4.6.9, for example.

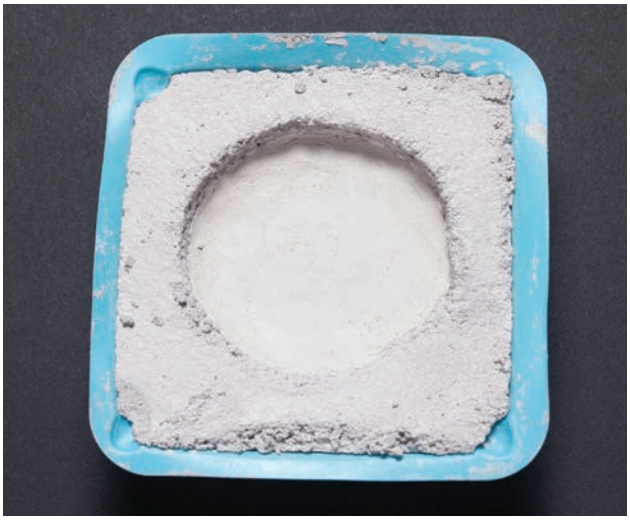


**Figure 4.6.6** Specimen exhibiting no drying shrinkage (NS).

**Figure 4.6.7** Specimen exhibiting medium drying shrinkage (MS).

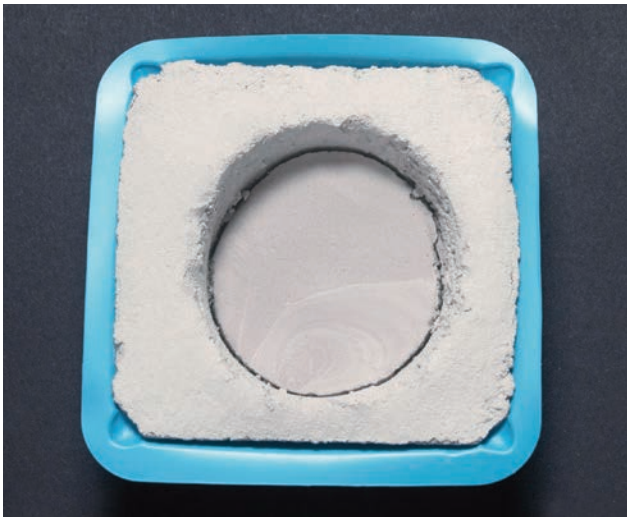


**Figure 4.6.8** Specimen exhibiting high drying shrinkage (HS).

## DRYING SHRINKAGE

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |

**NO DRYING SHRINKAGE (NS):** No separation between grout and mortar and no visible cracking in the grout

**MEDIUM DRYING SHRINKAGE (MS):** Separation of less than 0.5 mm between grout and mortar and/or a maximum crack size of less than 0.5 mm in the grout

**HIGH DRYING SHRINKAGE (HS):** Separation of more than 0.5 mm between grout and mortar and/or a maximum crack size of more than 0.5 mm in the grout

| Test 1 | | | | Test 2 | | | |
|---|---|---|---|---|---|---|---|
| **Date** | **Time** | **RH (%)** | **Temperature (°C)** | **Date** | **Time** | **RH (%)** | **Temperature (°C)** |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

| Test 1 | | | Test 2 | | |
|---|---|---|---|---|---|
| **Separation size (mm)** | **Crack size (mm)** | **Classification** | **Separation size (mm)** | **Crack size (mm)** | **Classification** |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

**Figure 4.6.9** Data collection sheet for the drying shrinkage test method.

## 4.7. Final Setting Time

### Aim

This test is used in the field to determine the final setting time of a grout. The time of setting is of interest particularly in situations where a fairly rapid set may be desired (e.g., fragile plasters, vaults, large areas of loss) or cases in which it is necessary to know when supports can be removed. Desirable setting time should be determined on a case-by-case basis, depending on the type of voids or cracks to be grouted.

### Description

This test defines the final setting time of a grout as the time at which it becomes rigid enough to resist penetration by a cannula. Grout is injected into a container, and the time of final setting is determined by periodic insertion of a cannula into the grout until the cannula can no longer penetrate it. During the measurement, the grout-filled containers are placed near the location where the grouting will be executed. Final setting time, as measured here, is different from the laboratory test for setting time with Vicat needle, as it takes into account the effects of drying due to a loss of water to the substrate and to evaporation. The results of this field test will be closer to those that will be observed in the field than those measured in the laboratory.

### Note

The experiment can be run by injecting grout directly into weighing boats or similar shallow trays, or by injecting grout into prepared "mortar cups" (described below). The second setup aims to address the effect of absorption by the substrate on final setting time, which is not taken into account if nonporous weighing boats or trays are used. The following procedure explains how to measure final setting time in mortar cups. Mortar used for repairs on site can be used to make mortar cups. The following mortar proportions and ingredients, given here as a reference, were used at the GCI to prepare these cups.

### Equipment and Materials

- standard sand (EN 196-1)
- pozzolan
- lime putty
- mixing bowl or large plastic container for mixing the mortar
- mortar mixer or trowel
- spatula
- plastic tube approximately 50 mm in diameter
- two weighing boats or similar shallow 30–50 mm deep containers at least 20 mm larger in diameter than the plastic tube described above
- 20 mL syringe
- 60 mL syringe
- 14 gauge stainless steel blunt cannula (dispensing needle) (outer diameter, 2.1 mm; inner diameter, 1.70 mm; length, 25.4 mm)
- 100 g granular material (2 mm minimum aggregate size)
- wristwatch
- thermometer
- humidity gauge
- plastic bags or moist cabinet

### Preparation of Mortar Cups

Prepare a minimum of two mortar cups per grout as follows:

1. Pour 2.5 parts (by volume) standard sand and 0.5 parts (by volume) pozzolan into a container and mix with a mortar mixer or trowel.
2. Add 1 part (by volume) lime putty and mix thoroughly with the mortar mixer at low speed, or mix manually with a trowel.
3. Pour the mortar mixture into a small plastic weighing boat or container, fill approximately 30 mm deep, and smooth the surface with a spatula (fig. 4.7.1).
4. Press one end of the plastic tube halfway (approx. 15 mm deep) into the mortar (not all the way to

**Figure 4.7.1** A weighing boat or shallow container filled with mortar.

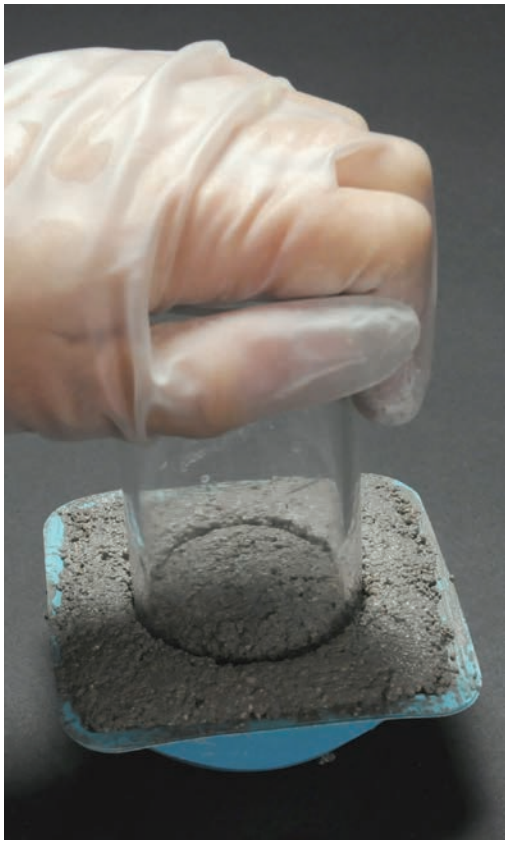


**Figure 4.7.2** Plastic tube being pressed and rotated to approximately half the depth of the mortar in the cup.

the bottom), and rotate it to hollow out a cylindrical cavity in the mortar (fig. 4.7.2).

5. Remove the plastic tube and remove excess mortar from inside the cylindrical cavity with the spatula to create a flat bottom (fig. 4.7.3). Reuse the plastic tube to prepare as many mortar cups as needed.
6. Store the mortar cups (fig. 4.7.4) in tightly closed plastic bags (or in a moist cabinet at RH greater than 95%) at 20°C ± 10°C for 2 weeks.
7. At the end of 2 weeks, remove the mortar cups from the plastic bags (or from the moist cabinet) and keep them near the location where the grouting will be conducted for at least 2 more weeks.

## Procedure

1. Using a syringe without a cannula, inject 20 mL grout into a plastic container or mortar cup cavity (fig. 4.7.5).

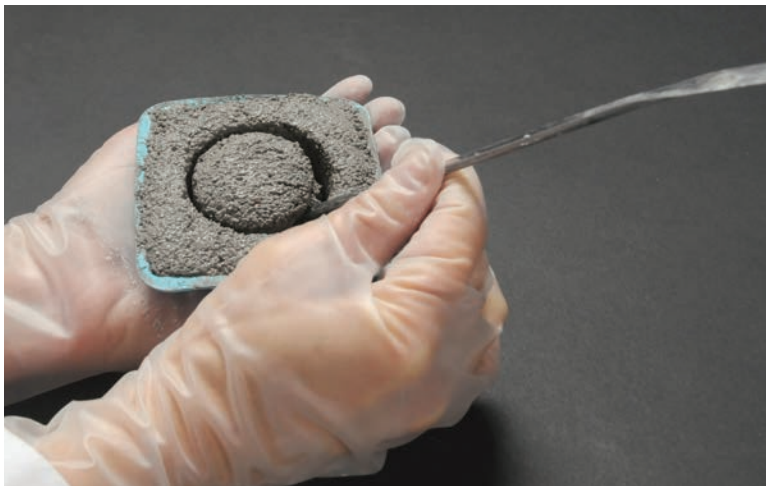


**Figure 4.7.3** Removing excess mortar from the cavity.

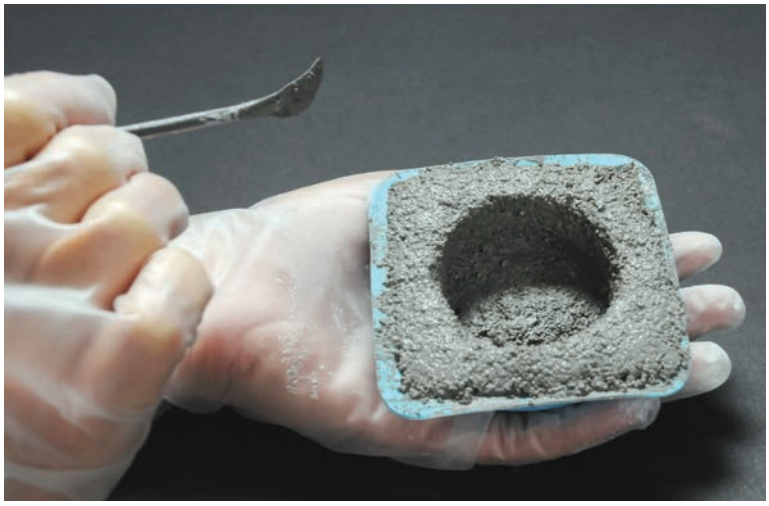


**Figure 4.7.4** Fresh mortar cup.

**Figure 4.7.5** Grout being injected into a mortar cup.

2. Tap the sides of the container 10 times to spread the grout, and remove any entrapped air by slowly hitting the container on the counter.
3. Prepare a second specimen following steps 1 and 2; a total of two specimens per grout are needed.
4. Record the time ($t_o$).
5. Leave the grout-filled plastic containers or mortar cups next to the area where the grouting will take place. Record the RH and ambient temperature.
6. Fill the 60 mL syringe (without a plunger) with 100 g granular material and attach the cannula.
7. After 1 hour, while holding the syringe vertically, position the tip of the cannula as close as possible to the surface of the grout specimen without touching it. The location of the first penetration hole should be at least 10 mm away from the wall of the mortar cup. Release the syringe and, using your fingers, guide it to fall directly into the grout (fig. 4.7.6).
8. Record the RH and temperature to monitor the effect of evaporation on final setting.
9. Repeat steps 7 and 8 every hour until the cannula no longer penetrates the grout (fig. 4.7.7). The space between two penetration holes should be at least 10 mm. The cannula should be cleaned, both inside and outside, after each measurement.

**Figure 4.7.6** Syringe with cannula filled with 100 g of sand being dropped into the grout.

**Figure 4.7.7** Final setting, reached after five penetrations.

### Results

1. Final setting time is the time when the cannula no longer penetrates the grout.
2. The average of two independent measurements is the average final setting time obtained under field conditions. Two measurements should not differ from each other by more than 2 hours; otherwise, the test should be repeated.
3. Record results, including all temperature and humidity measurements, on a data collection sheet; see figure 4.7.8 for an example.

**FINAL SETTING TIME**

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |

$t_0$: Time the specimen is prepared

$t_{final}$: Time at which the cannula does not penetrate grout specimen

| | Time (t) | | | Ambient | |
|---|---|---|---|---|---|
| | **Date** | **Hour** | **Min** | **Temperature (°C)** | **RH (%)** |
| ($t_0$) | | | | | |
| **Specimen 1** | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| ($t_0$) | | | | | |
| **Specimen 2** | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

| Specimen no. | $t_{final}$ (h) |
|---|---|
| 1 | |
| 2 | |

$t_{final}$, average = _____ h

**Figure 4.7.8** Data collection sheet for the final setting time test method.

## 4.8. Capillary Water Absorption

### Aim

This test is used to estimate the water absorption behavior of hardened grout under field conditions by a gravimetric method. The absorption of water in a hardened grout should correspond to that of the materials being grouted to ensure compatibility of original and intervention materials.

### Description

The method uses the sample size requirements of NORMAL 11/85—Capillary Water Absorption and Capillary Absorption Coefficient—and the procedure of RILEM test no. II.6—Water Absorption Coefficient (Capillarity)—and it follows the laboratory procedure for capillary water absorption (section 2.7 in this volume), with the exception of the processes of specimen preparation and oven drying, which have been altered for field testing. A syringe with a cannula is used to fill an empty transparent plastic tube through a taped, sealed end. The plastic tube is held vertically, and grout is injected through the tape into the bottom of the tube. After curing, the grout specimens are pushed out of the tube, or removed by cutting the plastic tube with a saw. Specimens are set on a perforated stand placed in a tray filled with water. The weight change of the specimen over time is used to calculate the amount of absorbed water.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces in the Field

One of the most important modifications to this test method is that the specimens are not dried in the oven at 40°C ± 1°C, as was the case for the laboratory test method. Therefore, obtained $A_{field}$ is to be used only for comparing water absorption capacity of the grouts tested at the same time under the same field conditions. The selected shape (cylindrical) and size (22 mm in diameter and 100 mm in height) of the grout specimens are meant to minimize cracking of specimens during curing, demolding, and drying. The selected dimensions are also suitable for testing brittle and heterogeneous injection grouts by providing a large enough sample volume and a surface area to volume ratio of 2.0 (NORMAL 11/85).

### Equipment and Materials

- plastic container with a surface area more than 20 times larger than the inflow surface area of the specimen and deep enough to cover the perforated stand and the upright specimen
- plastic lid fitting the container
- distilled water
- perforated stainless steel metal or plastic stand
- balance accurate to within 0.01 g
- transparent rigid plastic (e.g., polymethyl methacrylate, PMMA) tube with 22.00 ± 0.05 mm inner diameter and around 1.75 mm wall thickness
- adhesive tape
- 60 mL syringe
- 14 gauge stainless steel blunt cannula (dispensing needle) with outer diameter, 2.1 mm; inner diameter, 1.70 mm; length, 25.4 mm
- ruler in millimeters
- stopwatch
- lint-free cloth
- tile or manual saw with a PMMA cutting blade or PMMA cutting knife
- V-shaped holder (metal or wood)

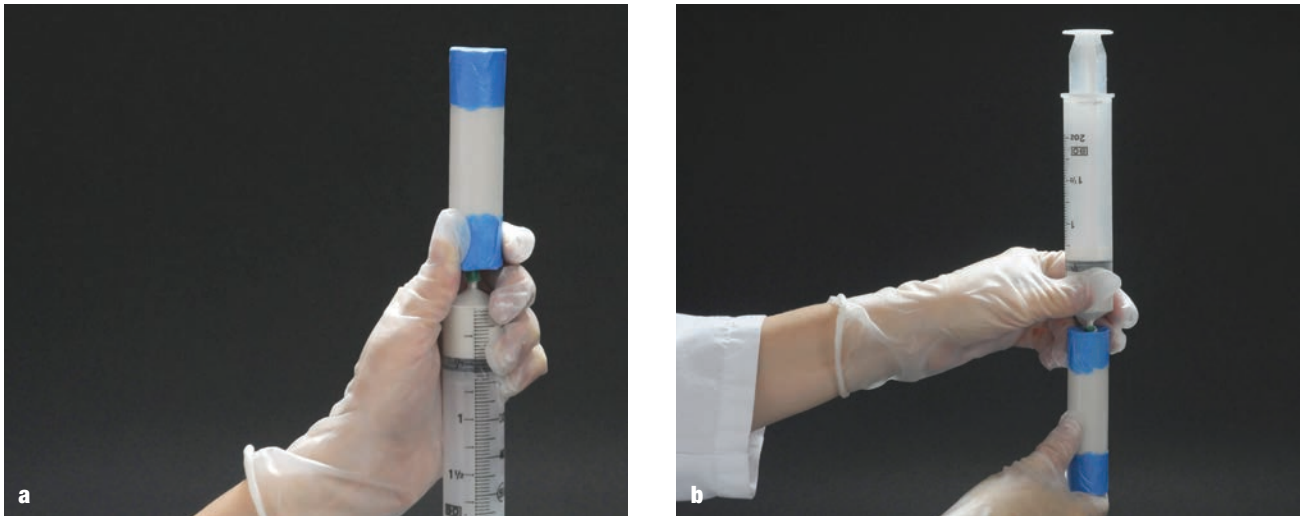
### Column Preparation

1. Cut transparent rigid plastic tube into columns 100 mm in length.
2. Prepare three columns for each grout.
3. Seal one end of the columns with tape.

### Specimen Preparation

1. Prepare the grout.
2. Pull 50 mL grout into the syringe and remove air bubbles by tapping with your finger.
3. Attach the cannula to the syringe and insert the cannula through the taped bottom end of the column (fig. 4.8.1).

**Figure 4.8.1** The column is filled with a syringe inserted from the taped bottom of the column.

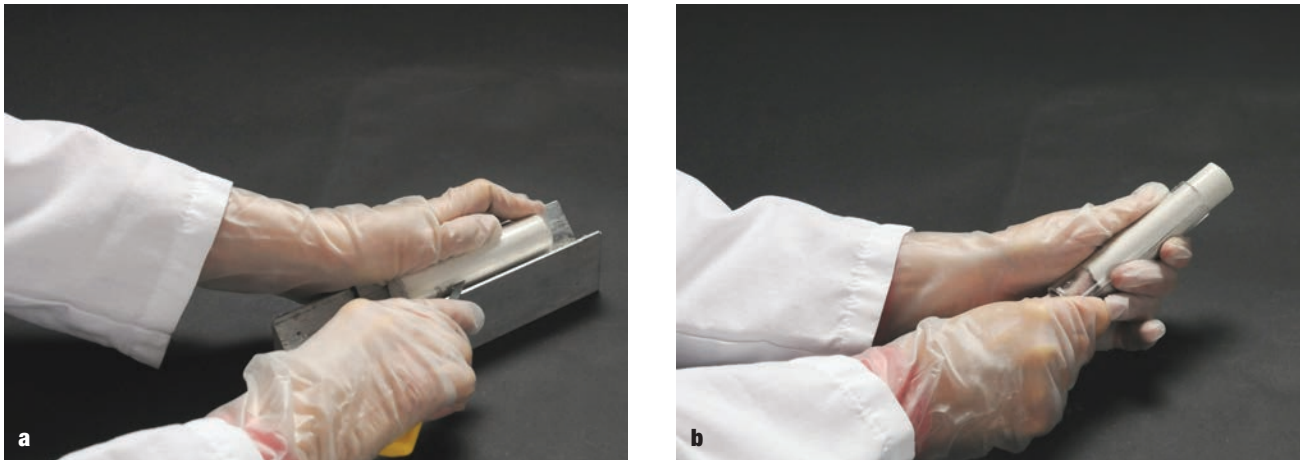



**Figures 4.8.2a and 4.8.2b** The top of the column sealed with adhesive tape (a), and the column turned upside down (b).

4. Push the plunger; holding the column vertically, slowly fill the column to the top with grout.
5. Seal the top end of the column with adhesive tape (fig. 4.8.2a). Turn the column upside down, remove the cannula, and reseal the end with fresh tape (fig. 4.8.2b).
6. Prepare three specimens for each grout.
7. Store the columns vertically (injection end down) for at least 2 weeks.
8. Extrude each specimen from the column by sawing the plastic tube covering the cylindrical specimen along the length without making any saw marks on the specimen (fig. 4.8.3a) and simply push the specimen with a finger from one end. Pushing the specimen with a finger from one end without sawing the plastic tube may also work (fig. 4.8.3b).

### Procedure

1. Place the specimens near the location where the grouting will be conducted and leave for 2 weeks. Record the maximum and minimum RH and the ambient temperature weekly.
2. Measure the length (l) and diameter (d) of each specimen.
3. Weigh the specimen to the nearest 0.01 g ($M_0$).
4. Fill the tray with distilled water until the water level is 2 mm above the perforated stand. During testing, the water level in the tray is kept constant by the addition of water as needed.
5. Place the specimen on the stand and start the stopwatch (fig. 4.8.4).
6. Stop the stopwatch after 30 seconds and simultaneously remove the specimen from the stand. Lightly blot the wet face with a damp cloth to remove




**Figures 4.8.3a and 4.8.3b** Slitting of a cylindrical plastic tube with a PMMA cutter (a), and removing the cured grout specimen (b).

**Figure 4.8.4** Specimen absorbing water after being placed in a water-filled container. The level of water absorbed is indicated by the darker area at the bottom of the grout specimen.

**Figure 4.8.5** Test setup before covering with a lid. The cover minimizes evaporation of the water in the tray.

surface water, and weigh the specimen ($M_t$) with the wet surface at the top. Place the specimen back on the stand as quickly as possible; start the stopwatch again.

7. Take measurements at 1, 2, 5, 10, 15, and 30 minutes.
8. The test setup is covered with a plastic lid to control the RH of the surroundings (fig. 4.8.5).

### Results

1. The amount of absorbed water after time $t$ ($\Delta M_t$), in grams, is calculated with

$$\Delta M_t = M_t - M_0 \tag{4.8.1}$$

where $M_t$ is the weight of the specimen at time $t$ in grams, and $M_0$ is the weight of the specimen at $t = 0$ in grams.

2. The weight of water absorbed per unit area (m) in kilograms per square meter is calculated with

$$m = \frac{\Delta M_t}{\pi \times \left(\frac{d}{2}\right)^2} \times 10^3 \tag{4.8.2}$$

where $\Delta M_t$ is the amount of absorbed water after time $t$, in grams, and $d$ is the diameter of the grout specimen in millimeters.

3. Calculate the water absorption coefficient in the field, $A_{field}$, in kilograms per square meter per square root of seconds ($kg \cdot m^{-2} \cdot s^{-1/2}$) using the results at 1 minute and 15 minutes, as follows:

$$A_{field} = \frac{m_{15min} - m_{1min}}{\sqrt{t_{15min}} - \sqrt{t_{1min}}} = \frac{m_{15min} - m_{1min}}{\sqrt{900} - \sqrt{60}} \tag{4.8.3}$$

where $m_{15min}$ is the weight of water absorbed per unit area at 15 minutes, and $m_{1min}$ is the weight of water absorbed per unit area at 1 minute, both in kilograms per square meter. The value $t_{15min}$ is the time at 15 minutes, and $t_{1min}$ is the time at 1 minute, both in seconds.

4. Calculate the average water absorption coefficient using the results of three specimens. Discard any individual result if the average and the individual result differ more than 25% from each other. The average water absorption coefficient should be obtained by at least two individual results; otherwise, the test should be repeated.
5. An example data collection sheet is given in figure 4.8.6.

## CAPILLARY WATER ABSORPTION

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |

**l (mm):** Length of the specimen
**d (mm):** Diameter of the specimen
**$M_0$ (g):** Weight of the specimen at time t = 0
**t:** Time
**$M_t$ (g):** Weight of the specimen at time t
**$\Delta M_t$ (g):** Weight of absorbed water after time t
**m (kg·m$^{-2}$):** Weight of absorbed water per unit area
**$A_{field}$ (kg·m$^{-2}$·s$^{-1/2}$):** Water absorption coefficient measured in the field

### EQUATIONS

$$\Delta M_t = M_t - M_0 \qquad m = \frac{\Delta M_t}{\pi \times \left(\frac{d}{2}\right)^2} \times 10^3$$

$$A_{field} = \frac{m_{15\ min} - m_{1\ min}}{\sqrt{t_{15\ min}} - \sqrt{t_{1\ min}}} = \frac{m_{15\ min} - m_{1\ min}}{\sqrt{900} - \sqrt{60}}$$

| Specimen no. | l (mm) | d (mm) | $M_0$ (g) |
|---|---|---|---|
| 1 | | | |
| 2 | | | |
| 3 | | | |

| | Temperature (°C) | | RH (%) | |
|---|---|---|---|---|
| Week no. | max | min | max | min |
| 1 | | | | |
| 2 | | | | |

| | | Specimen 1 | | | Specimen 2 | | | Specimen 3 | | |
|---|---|---|---|---|---|---|---|---|---|---|
| t (min) | t (s) | $M_t$ (g) | $\Delta M_t$ (g) | m (kg·m$^{-2}$) | $M_t$ (g) | $\Delta M_t$ (g) | m (kg·m$^{-2}$) | $M_t$ (g) | $\Delta M_t$ (g) | m (kg·m$^{-2}$) |
| 0.5 | 30 | | | | | | | | | |
| 1 | 60 | | | | | | | | | |
| 2 | 120 | | | | | | | | | |
| 5 | 300 | | | | | | | | | |
| 10 | 600 | | | | | | | | | |
| 15 | 900 | | | | | | | | | |
| 30 | 1800 | | | | | | | | | |

| Specimen no. | $A_{field}$ (kg·m$^{-2}$·s$^{-1/2}$) |
|---|---|
| 1 | |
| 2 | |
| 3 | |

**$A_{field}$, average =** _____ kg·m$^{-2}$·s$^{-1/2}$

**Figure 4.8.6** Data collection sheet for the capillary water absorption test method.

## 4.9. Water Vapor Transmission Rate by the Wet Cup Method

### Aim

This test estimates the passage of water vapor through hardened injection grouts under field conditions. The rate of water vapor transmission of a grout should be similar to the rate of water vapor transmission of the original material to be grouted, in order to ensure passage of water vapor through the original and intervention layers.

### Description

The method uses the sample size requirements of NORMAL 21/85—Water Vapor Permeability—and loosely follows the procedure of ASTM E 96—Standard Test Methods for Water Vapor Transmission of Materials—as well as following the laboratory procedure for water vapor transmission by the wet cup method (section 2.8 in this volume), with the exceptions of controlled environmental conditions and dry cup correction, which have been modified for the field. A grout specimen is sealed to the open mouth of a test cup partially filled with distilled water and placed in the area where the grouting will be carried out. Periodic weighings of the cup and specimen determine the rate of water vapor movement (i.e., water vapor transmission rate—$WVTR_{field}$) through the specimen from the inside of the cup to the atmosphere.

### Adaptations for Testing Injection Grouts for the Conservation of Architectural Surfaces in the Field

The specimen dimension requirements of NORMAL 21/85, including the requirements that the thickness be at least twice the size of the largest aggregate and the diameter be at least three times the thickness of the specimen, are followed. The selected shape (discs) and size (44 mm diameter and 10.0 mm thick) of the grout specimens are designed to minimize cracking of the specimen during curing and demolding. Furthermore, the disc shape simplifies the lateral sealing, and the selected dimensions ensure that the specimen is representative of the material being tested. One of the most important modifications applied to this test method is that the test is conducted under field conditions (i.e., in varying relative RH and temperature). Therefore, the obtained $WVTR_{field}$ is to be used only for comparing water vapor transmission rates of the grouts tested at the same time under the same field conditions. It is also recommended that RH and temperature ranges during the measurements be recorded.

### Notes

The experiment can be run by preparing specimens on nonporous (e.g., glass or tile) surfaces or on porous substrates (e.g., plastered tile, brick, or stone). The second setup takes into account the effect of water loss due to the absorption by the substrate on the water vapor transmission rate of grouts. To prepare the plastered tile, mortar used for repairs on site can be employed. The following proportions and ingredients were used at the GCI to prepare the plastered tile, and they are given here as a reference.

### Equipment and Materials

- standard sand (EN 196-1)
- superventilated pozzolana
- lime putty
- mixing bowl or large plastic container to mix the plaster
- mortar mixer, if available
- trowel
- glazed or unglazed ceramic tile (33 × 25 × about 1 cm thick)
- spray bottle
- distilled water
- three ring molds cut from rigid plastic tubing (approximately 44.0 mm inner diameter and 10.0 mm thick) (fig. 4.9.1)
- hot glue gun and glue sticks, or plasticine
- three plastic cups with maximum 50 mm mouth diameter (fig. 4.9.2)
- thin screen or cheesecloth
- 10 mL syringe
- sealing gum or cord (e.g., Permagum sealing cord)
- balance accurate to within 0.01 g
- metric caliper
- thermometer
- humidity gauge
- wristwatch
- plastic bag or moist cabinet larger than the size of the ceramic tile

### Preparation of Plastered Tile

1. Pour 2.5 parts (by volume) standard sand and 0.5 parts (by volume) superventilated pozzolana into a container and mix with mortar mixer or trowel.

2. Add 1 part (by volume) lime putty and mix thoroughly with the mortar mixer at low speed, or mix manually with a trowel.
3. Using the spray bottle, prewet the reverse of the tile with water.
4. Spread plaster over the reverse of the tile to a thickness of 3–4 cm.
5. Smooth the surface with the trowel.
6. Store the plastered tile in a tightly closed plastic bag (or in a moist cabinet at RH greater than 95%) at 20°C ± 5°C for 2 weeks.
7. At the end of 2 weeks, remove the plastered tile from the plastic bag (or from the moist cabinet) and keep it near the location where the grouting will be executed for at least 2 more weeks. Record the maximum and minimum RH and temperature weekly.

### Specimen Preparation

1. Place the mold on the plastered tile, brick, or stone covered with a thin screen (or cheesecloth), as shown in figure 4.9.1.
2. Hold the ring mold down and attach the bottom of the mold to the plastered tile by gluing it around the bottom edge with hot glue or plasticine.
3. Prepare a total of three molds for each grout.
4. Fill the ring molds with grout flush with the top within 2 minutes after mixing (fig. 4.9.1). Strike off the grout with the edge of a trowel.
5. Store the specimen assembly in a tightly closed plastic bag (or in a moist cabinet at RH greater than 95%) at 20°C ± 1°C for 2 weeks.
6. At the end of the first week, remove the specimen assembly from plastic bag (or from the moist cabinet). Carefully remove the hot glue around the mold and separate the specimens and the molds from the plaster by pulling the screen horizontally. Replace the specimen assembly in a tightly closed plastic bag (or in a moist cabinet) for one more week.
7. At the end of second week, remove the specimen assembly from plastic bag (or from the moist cabinet). Leave the specimens in the molds uncovered in the area where grouting will be conducted, out of direct sunlight and wind, for 24 hours before demolding.
8. Demold the specimens. First separate the specimen and the mold from the screen, if needed.
9. Keep specimens near the location where the grouting will be conducted for another 13 days, or until test is performed.
10. Record the maximum and minimum RH and temperature weekly.

### Procedure

1. Label three plastic cups for each grout.
2. Measure the diameter (d) and the thickness (δ) of the specimens.
3. Pour 10 mL distilled water into the cups.

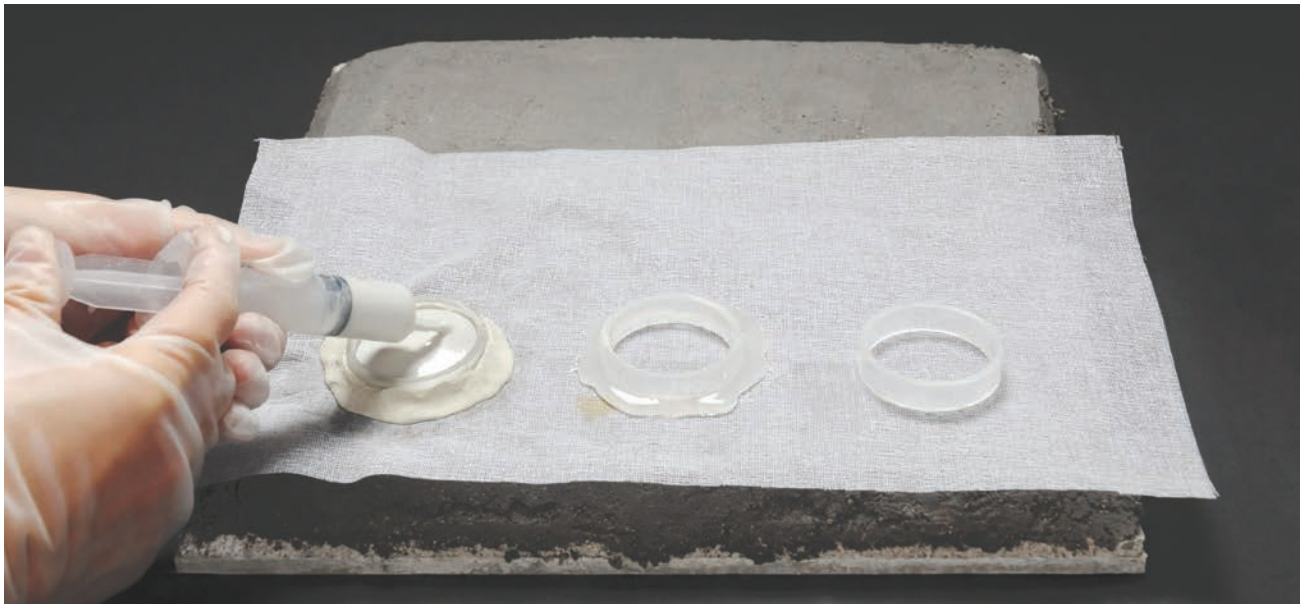


**Figure 4.9.1** Molds attached to plastered tile being filled with grout.

**Figure 4.9.2** Cup with cured specimen after sealing.

4. Place the sealing gum or cord around the specimen, and place the specimen in the cup. Gently push the specimen, being careful not to apply excessive pressure, so as to avoid cracking the specimen or pushing the sealing gum underneath the specimen.
5. Continue to fill the space between the grout sample and the cup with sealant to cover the sides of the specimen (fig. 4.9.2).
6. Weigh the cup and specimen assembly ($M_0$).
7. Place the cups near the location where the grouting will be conducted.
8. Weigh the cups (M) every day for 2 weeks. Record the RH and ambient temperature at each weighing.

## Results

1. Calculate the surface area of the specimen (a) in square meters using

$$a = \frac{\pi \times d^2}{4 \times 10^6} \qquad (4.9.1)$$

where $d$ is the diameter of the specimen in millimeters.

2. Calculate the weight loss (ΔM) of each specimen and cup assembly for each measurement, in grams, using

$$\Delta M = M - M_0 \qquad (4.9.2)$$

where $M$ is the weight of the specimen and cup, and $M_0$ is the initial weight of the specimen and cup, both in grams.

3. Calculate the rate of water vapor transmission ($WVTR_{field)}$ in grams per hour per square meter ($g \cdot h^{-1} \cdot m^{-2}$), using the results at 2 days and 14 days, as follows:

$$WVTR_{field} = \frac{\Delta M_{day\,14} - \Delta M_{day\,2}}{288 \times a} \qquad (4.9.3)$$

where $\Delta M_{day\,14}$ is the weight loss of the specimen and cup at 14 days, in grams, $\Delta M_{day\,2}$ is the weight loss of the specimen and cup at 2 days, in grams, and $a$ is the surface area of the specimen in square meters. The most common way of expressing WVTR is in grams per day (24 hours) per square meter ($g \cdot 24h^{-1} \cdot m^{-2}$). For the best use of $WVTR_{field}$ values, record the RH and the ambient temperature ranges during the measurement. The thickness of the specimens should also be indicated.

4. Calculate average $WVTR_{field}$ from the results of three specimens. Discard any result if the average and the independent result differ more than 25%. Average $WVTR_{field}$ should be obtained by at least two individual results; otherwise, the test should be repeated.
5. An example data collection sheet is given in figure 4.9.3.

## WATER VAPOR TRANSMISSION RATE BY THE WET CUP METHOD

| | |
|---|---|
| **Grout name** | |
| **Grout proportions** | |
| **Operator** | |
| **Date** | |

**d (mm):** Diameter of mouth area of the cup
**δ (mm):** Thickness of the specimen
**$M_0$ (g):** Initial weight of the the specimen and cup
**t:** Time
**M (g):** Weight of the specimen and cup at time t
**a ($m^2$):** Area of the specimen
**ΔM (g):** Weight loss of the specimen and cup assembly at time t
**$\Delta M_{day14}$ (g):** Weight loss of the specimen and cup at 14 days
**$\Delta M_{day2}$ (g):** Weight loss of the specimen and cup at 2 days
**$WVTR_{field}$ ($g \cdot h^{-1} \cdot m^{-2}$):** Rate of water vapor transmission

| | Week no. | Temperature (°C) Max | Temperature (°C) Min | RH (%) Max | RH (%) Min |
|---|---|---|---|---|---|
| Plaster | 1 | | | | |
| | 2 | | | | |
| Specimen | 1 | | | | |
| | 2 | | | | |

### EQUATIONS

$$a = \frac{\pi \times d^2}{4 \times 10^6} \qquad \Delta M = M_t - M_0$$

$$WVTR_{field} = \frac{M_{day\ 14} - M_{day\ 2}}{288 \times a}$$

| Specimen no. | d (mm) | δ (mm) | $M_0$ (g) | a ($m^2$) |
|---|---|---|---|---|
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |

| Time (t) Date | Time (t) Hour | Time (t) Min | Temp. (°C) | RH (%) | M (g) 1 | M (g) 2 | M (g) 3 | ΔM (g) 1 | ΔM (g) 2 | ΔM (g) 3 |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | |
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| | | | | | | | | | | |

| | Temp. (°C) | RH (%) |
|---|---|---|
| Maximum | | |
| Minimum | | |

| Specimen no. | $WVTR_{field}$ ($g \cdot h^{-1} \cdot m^{-2}$) |
|---|---|
| 1 | |
| 2 | |
| 3 | |

**$WVTR_{field}$, average =** _____ $g \cdot h^{-1} \cdot m^{-2}$

**Figure 4.9.3** Data collection sheet for the water vapor transmission rate test method.

# Appendix A: List of Standards



The following list of standard and recommended tests is meant as a reference for the different tests that were modified for this manual. Some laboratory procedures described here closely follow one or another standard. Others loosely follow a given standard, and still others were derived from two or more standards and/or recommendations, and modified according to different material properties and performance characteristics of injection grouts from those materials for which these tests were developed.

The test methods are designated by the following acronyms, which specify either the organizations that promulgated the standards (e.g., ASTM, DIN, EN, UNI, etc.) or a specific category of testing recommendations offered by an organization (e.g., RILEM, NORMAL, etc.).

| | |
|---|---|
| ASTM | ASTM International (formerly American Society for Testing and Materials) |
| DIN | Deutsches Institut für Normung (German Institute for Standardization) |
| EN | European standard approved by European Committee for Standardization (CEN) |
| NORMAL | Commissione Normativa Manufatti Lapidei (NORMAL) (Italian Committee for Normalization of Procedures on Stone Materials) |
| RILEM | Réunion Internationale des Laboratoires d'Essais et de Recherches sur les Materiaux et des Constructions (International Union of Testing and Research Laboratories for Materials and Structures) |
| UNI | Ente Nazionale Italiano di Unificazione (Italian Organization for Standardization) |

| | |
|---|---|
| **ASTM C 185** | Standard Test Method for Air Content of Hydraulic Cement Mortar |
| **ASTM C 191** | Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle |
| **ASTM C 192-07** | Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory |
| **ASTM C 474** | Standard Test Methods for Joint Treatment Materials for Gypsum Board Construction |
| **ASTM C 496** | Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens |
| **ASTM C 939** | Standard Test Method for Flow of Grout for Preplaced-Aggregate Concrete (Flow Cone Method) |
| **ASTM C 940** | Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory |
| **ASTM C 953** | Standard Test Method for Time of Setting of Grouts for Preplaced-Aggregate Concrete in the Laboratory |
| **ASTM C 1506** | Standard Test Method for Water Retention of Hydraulic Cement-Based Mortars and Plasters |
| **ASTM D 905** | Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading |
| **ASTM D 4327** | Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography |

| | |
|---|---|
| **ASTM E 96** | Standard Test Methods for Water Vapor Transmission of Materials |
| **DIN 18 555, part 7** | Testing of Mortars Containing Mineral Binders: Determination of Water Retentivity of Freshly Mixed Mortar by the Filter Plate Method to Determine the Water Retention Value |
| **EN 196-1** | Methods of Testing Cement—Part 1: Determination of Strength |
| **EN 445** | Grout for Prestressing Tendons—Test Method |
| **EN 1771** | Determination of Injectability Using the Sand Column Test |
| **NORMAL 11/85** | Capillary Water Absorption and Capillary Absorption Coefficient |
| **NORMAL 21/85** | Water Vapour Permeability |
| **RILEM test no. II.6** | Water Absorption Coefficient (Capillarity) |
| **UNI 11152** | Water Suspensions of Hydraulic Binders for Grouting: Characteristics and Test Methods |

# Appendix B: Laboratory Instruments and Manufacturers



| Company name, type (model) | Address | Telephone | Website |
|---|---|---|---|
| **Caframo**<br>Mixer<br>(Stirrer BDC 3030) | 501273 Grey Road 1<br>RR 2, Wiarton, Ontario, N0H 2T0,<br>Canada | +1 800 567 3556 | www.caframo.com |
| **Dionex**<br>Ion chromatograph, autosampler<br>(DX 500, AS 3500) | 1228 Titan Way, P.O. Box 3603,<br>Sunnyvale, CA 94088-3603,<br>USA | +1 408 737 0700 | www.dionex.com |
| **Humboldt Manufacturing Co.**<br>Water retention apparatus<br>(H-3630A) | 3801 North 25th Ave.,<br>Schiller Park, IL 60176,<br>USA | +1 408 737 0700 | www.humboldtmfg.com |
| **IGM**<br>Injectability apparatus<br>(I07 760) | 4, rue Pablo Picasso,<br>78114 Magny les Hameaux,<br>France | +33 1 30 47 10 65 | www.igm.fr |
| **IKA**<br>Flat stirrer<br>(KS250 basic) | 2635 North Chase Pkwy. SE,<br>Wilmington, NC 28405-7419,<br>USA | +1 910 452 7059 | www.ika.com |
| **Instron**<br>Universal testing machine<br>(5885H) | 825 University Ave.,<br>Norwood, MA 02062-2643,<br>USA | +1 800 877 6674 | www.instron.us |
| **Julabo**<br>Refrigerated/heating circulator<br>(F25) | 884 Marcon Blvd,<br>Allentown, PA 18109,<br>USA | +1 610 231 0250 | www.julabo.de/de |
| **Matest**<br>Cement setting tester<br>(Vicatronic) | Via delle Industrie, 25,<br>24048 Treviolo (Bg),<br>Italy | +39 035 2055011 | www.matest.com |
| **MK Diamond Products**<br>Tile saw<br>(MK 101) | 1315 Storm Pkwy.,<br>Torrance, CA 90501,<br>USA | +1 800 421 5830 | www.mkdiamond.com |
| **Thermo**<br>Viscosity tester<br>(Haake Viscotester 550) | 81 Wyman St.,<br>Waltham, MA 02454,<br>USA | +1 781 622 1000 | www.thermofisher.com |

# Glossary

The following terms have been used throughout this manual. They were taken from different sources describing related materials, parameters, and test methods.

The following publications were consulted in the preparation of this glossary: *Use of Nomenclature in Dispersion Science and Technology* by V. A. Hackley and C. F. Ferraris, National Institute of Standards and Technology (NIST) Special Publication 960-3 (Springfield, VA: NIST, 2001); and *Concrete,* 2d ed., edited by S. Mindess, J. F. Young, and D. Darwin (Upper Saddle River, NJ: Prentice Hall, 2003). Also consulted were the following standards from ASTM International: ASTM C 11, Standard Terminology Relating to Gypsum and Related Building Materials and Systems; ASTM C 125, Standard Terminology Relating to Concrete and Concrete Aggregates; and ASTM C 219, Standard Terminology Relating to Hydraulic Cement.

A selection of definitions has been reprinted with permission from *Glossary of Terms and Definitions Used in Grouting: Proposed Definitions and Preferred Usage,* Project Report 61 (London: CIRIA, 1997), ISBN: 978-0-86017-861-3; www.ciria.org.

**Adhesion**
The tendency of dissimilar materials and/or surfaces to bond to one another.

**Aliquot**
A small quantitative sample taken from a larger volume of a solution.

**Apparent Bingham yield stress**
A critical shear stress value of a nonideal Bingham plastic fluid, defined as the extrapolation of the flow curve from the linear, high shear rate region (plastic region) to the stress axis (shear rate of 0).

**Batch**
A quantity of material (e.g., grout) mixed at one time.

**Bingham plastic fluid**
A fluid that behaves as a solid at low stresses and as a viscous fluid at high stresses and exhibits rheological properties based on initial yield stress and plastic viscosity.

**Bleeding**
The separation of mixing water within, or its emergence from, a newly placed suspension (e.g., grout), caused by the settlement of the solid materials of the suspension.

**Bond strength**
The relative resistance to sliding or separation of a material (e.g., grout) from another material with which it is in contact. It is a collective expression of the effect of all forces between two surfaces, such as adhesion, friction, and mechanical interlock.

**Cannula**
A small-diameter metal or plastic tube (e.g., a hypodermic needle) for insertion into a cavity, as for draining off fluid or introducing medication. Used in these procedures for the injection of fresh grouts, for prewetting of surfaces, and for draining off surface water in some test procedures.

**Capillary water absorption**
The quantity of water absorbed by a porous material (e.g., hardened grout) in contact with a free water surface, by capillary action.

**Capillary water absorption coefficient**
The mass of water absorbed per unit surface area per square root of time. It corresponds to the slope of the initial straight segment of the capillary absorption curve.

**Capillary water absorption curve**
A curve that shows the change in the mass of water absorbed per unit surface area of a porous material (e.g., hardened grout), as a function of the square root of time under standard conditions.

**Consistency**
A qualitative expression of the relative mobility or ability of a freshly mixed suspension (e.g., grout) to flow.

**Curing**
For a lime-based grout, the maturing of the material by the hydration of its cementitious and/or pozzolanic materials and/or by the carbonation of lime components.

**Curing time**
For a grout, the time interval between the mixing of ingredients and the development of the ultimate solid properties of the material.

**Differential viscosity**
The derivative of shear stress with respect to shear rate.

**Drying shrinkage**
A reduction in dimensions of a material (e.g., grout) caused by drying.

**Eluent**
The mobile phase that transports the chemical through the column of chromatography.

**Expansion**
The increase in the volume of a suspension (e.g., grout), generally expressed as a percentage of the original volume of the material.

**Filtration**
The process of separating solids from fluids (liquids or gases) by placing a porous material (such as a filter) through which only the fluid can pass. The filter retains the suspended solid

particles, a process that may result in the deposition of a filter cake.

**Final bleed water**
The maximum percentage of the water phase released from a suspension (e.g., grout) during gravity settling, expressed as a percentage of the original volume.

**Final set**
A degree of stiffening (e.g., of a grout) greater than the initial set, generally stated as an empirical value indicating the time, in hours and minutes, that is required for stiffening that is sufficient to resist the penetration of a weighted test needle.

**Flow cone**
A device for measuring the consistency of a grout by measuring the time of efflux of a predetermined volume of grout to flow through a precisely sized orifice from a cone with standardized dimensions, used to indicate consistency.

**Flow curve**
A graphic representation of the behavior of flowing materials, in which shear stress is plotted as a function of shear rate.

**Grout**
A fluid material used to fill cracks, crevices, and cavities in soil, rock formations, and architectural and structural systems by force of gravity, injecting, or flooding, which gels, stiffens, or sets with time and consolidates the soil, rock, or wall, and/or reattaches layers in the architectural system. See "injection grout for architectural surfaces."

**Grout mix**
The components of a grout mixture, including dry ingredients (binders, aggregates, pozzolans, etc.) and water, expressed by weight.

**Hardening**
The development in a material of useful and measurable strength after setting.

**Hydration**
The chemical reaction between the hydraulic compounds and water (e.g., in a grout) that results in the formation of new compounds, most of which have strength-producing properties.

**Hydraulic binder**
A finely ground inorganic material or a blend of finely ground inorganic materials that when mixed with water sets and hardens, even under water, by means of hydration and/or pozzolanic reactions.

**Initial set**
A degree of stiffening (e.g., of a grout) generally stated as an empirical value indicating the time, in hours and minutes, that is required for stiffening that is sufficient to resist a predetermined penetration depth of a weighted test needle.

**Injectability**
The ability of a grout, under constant pressure, to fill a crack or capillary network created by granular material.

**Injectability curve**
The curve obtained by plotting the distance attained by a grout into a column filled with granular material as a function of time during an injectability test.

**Injection grout for architectural surfaces**
A bulked fluid material, composed of a binder, aggregate, water, and frequently admixtures, that can be injected behind a plaster, wall painting, or mosaic to fill cracks and voids and reestablish adhesion between delaminated layers.

**Ion chromatography**
A form of liquid chromatography, in which ions and polar molecules are separated based on their charge during their transport through a column, most commonly through ion exchange. After separation, each substance is quantified by a suitable means of detection.

**Laminar flow**
Flow across a surface in which all of the fluid particles proceed along parallel paths at the same velocity, and in which there is no transverse component of velocity.

**Marsh funnel**
See "flow cone."

**Maximum aggregate size**
The smallest sieve opening through which the entire sample (i.e., dry grout) can be passed.

**Mortar**
A combination of one or more binders (earth, gypsum, lime, or cement), aggregates, and water, with or without admixtures and fibrous reinforcements, used in or applied to masonry. In comparison with injection grouts, mortars typically have higher viscosity, contain aggregates of greater particle size, and have lower binder-water ratios.

**Newtonian fluid**
An ideal fluid that exhibits constant viscosity at all rates of shear.

**Non-Newtonian fluid**
A fluid that exhibits varying viscosity with different rates of shear and exhibits yield stress. See also "Bingham fluid."

**Particle size distribution**
A listing of percentages of all particle sizes in a sample, as determined by sieve analysis of dry granular materials for large particles, and determined by laser refractometry or sedimentation test for a suspension of small particles in a fluid. When measured by sieve analysis, it is usually expressed in terms of cumulative weight percentages of larger particles (retained) or smaller particles (passed).

**Plastic viscosity**
A parameter of the Bingham plastic model of fluids, the differential viscosity in the linear region of a flow curve—that is, the slope of the shear stress/shear rate curve above the yield point.

**Preplaced-aggregate concrete**
Concrete produced by placing coarse aggregate within a form or shutter and injecting a cementitious or resin grout to fill the interstices between the aggregate particles.

**Rheology**
The study of the deformation and flow of matter.

**Rotary viscometer**
An instrument for measurement of the viscosity of a fluid from the relationship between torque needed to rotate an

object submerged in fluid and rotation speed (shear gradient). The viscosity is derived from the ratio of shear stress related to torque to shear rate related to rotation speed.

**Sedimentation**
The accumulation of particles in a suspension at the bottom of a container as a result of gravitational forces.

**Separator column**
An ion exchange column used to separate ions in a solution according to their retention characteristics on the column packing material prior to quantification.

**Set**
A change of phase from a fluid to a solid. For a suspension, this condition is reached when it has lost plasticity to an arbitrary degree (i.e., the onset of rigidity), usually measured in terms of resistance to penetration or deformation; *initial set* refers to the first stiffening, and *final set* refers to an attainment of significant rigidity.

**Setting time**
The elapsed time required for a material to attain a specified rigidity after its components are mixed.

**Shear**
The relative movement resulting from the application of forces that causes, or tends to cause, two contiguous parts of a material to slide relative to one another in a direction parallel to their plane of contact.

**Shear bond strength**
The stress at which the bond between two different materials can no longer resist an applied shear force, and failure occurs.

**Shear rate**
The rate at which shear is applied, as a function of time.

**Shear strength**
The stress at which a single material can no longer resist an applied shear force, and failure occurs.

**Shear stress**
The component of an applied stress that causes successive parallel layers of a material body to move, in their own planes (i.e., the plane of shear), relative to one another.

**Shrinkage**
A reduction in volume of a material.

**Splitting tensile strength test (Brazilian test)**
An indirect way of measuring the tensile strength of a material. A load is applied on the side of a cylindrical specimen in diametric compression, leading to splitting of the specimen across the vertical diameter.

**Stress**
Force per unit area of a surface in a specimen on which internal forces act.

**Suspension**
A heterogeneous fluid containing solid particles that are either sufficiently large and settle due to gravitational forces, or small enough that they remain suspended in the fluid (colloidal particles).

**Tensile strength**
The stress at which a material can no longer resist an applied tensile force, and failure occurs.

**Thixotropy**
A reversible, time-dependent decrease in viscosity at a particular shear rate.

**Turbulent flow**
Flow in which the progression of fluid particles is irregular, and there is a seemingly haphazard interchange of particle positions. Individual particles are subject to fluctuating transverse velocities, so that the motion is eddying and sinuous rather than rectilinear.

**Viscometer**
An instrument for measuring the viscosity of fluids.

**Viscoplastic**
A hybrid property of a material that behaves like a solid below some critical stress value (the yield stress) but flows like a viscous liquid when this stress is exceeded. It is often associated with highly aggregated suspensions.

**Viscosity**
The property of a fluid that resists the forces tending to make it flow.

**Water retention capacity**
The ability of a suspension (i.e., freshly mixed grout) to retain its water under suction created by contact with a porous body.

**Water retention value**
The mass of water retained in a suspension after a specified amount of suction is applied for a specified period of time. Expressed as a percentage of the original water content.

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

**Water vapor transmission rate**
The steady water vapor flow through a porous material (e.g., grout) in unit time through unit area, normal to specific parallel surfaces, under specific conditions of temperature and humidity at each surface.

**Wet density**
The weight per unit volume of a freshly mixed suspension (e.g., grout).

**Yield stress**
A critical shear stress value below which an ideal plastic or viscoplastic material behaves like a solid, and above which a plastic material yields (i.e., deforms plastically), while a viscoplastic material flows like a liquid.

# Contributors

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**Leslie Rainer** is a wall paintings conservator and a senior project specialist at the Getty Conservation Institute, where she manages the Injection Grouts for the Conservation of Architectural Surfaces project and other projects related to wall paintings conservation. She received an independent master's degree in the conservation of decorated architectural surfaces from Antioch University and a certificate from the ICCROM Mural Paintings Conservation course. She is a recipient of the Rome Prize in Conservation and Historic Preservation and has published in professional journals. Rainer is a member of AIC, IIC, ICOMOS, ICOM-CC, and WAAC.

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