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# Field test methods for comparative evaluation of lime-based hydraulic injection grouts for the conservation of architectural surfaces

**BERIL BIÇER-ŞIMŞIR***
Getty Conservation Institute
Los Angeles CA, USA
bbicersimsir@getty.edu

**LESLIE RAINER**
Getty Conservation Institute
Los Angeles CA, USA
lrainer@getty.edu

*Author for correspondence

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## ABSTRACT

This paper discusses field tests developed by the Getty Conservation Institute to evaluate injection grouts for comparison and quality control of materials and mixtures. Field test methods discussed include injection with syringe, flow with syringe, expansion and bleeding, wet density, drying shrinkage, and final setting time. Four commercial grouts and an ICCROM grout with four modified versions were tested to demonstrate the use, effectiveness, and limitations of these field tests.

## INTRODUCTION

Injection grouts for the conservation of architectural surfaces were developed 30 years ago as a means of preserving plasters, wall paintings, and mosaics in situ (Ferragni et al. 1984), and are widely used in current conservation practice. Since the first injection grouts developed at ICCROM, a number of commercial grouts and custom-mixed grouts have been developed and are used for the in-situ conservation of architectural surfaces in the field. The optimal performance of these materials to ensure durable treatments is highly dependent on the selection of the appropriate grout for specific cases and conditions. However, the lack of suitable standard tests leading to the use of a wide range of different test methods by manufacturers and researchers makes it difficult to compare properties and presents challenges to practitioners in their selection of grouts. Finally, limited systematic research is available, allowing conservators to evaluate and compare grouts for specific applications (Biçer-Şimşir et al. 2009).

In order to address these issues, the Getty Conservation Institute (GCI) has undertaken an interdisciplinary project with scientists and conservators on the evaluation of injection grouts for the stabilization of architectural surfaces. Supported by laboratory and field test methods, the project has aimed to develop a methodology (Figure 1) for the evaluation and selection of injection grouts which were specifically designed for these materials. Following preliminary background research on the topic, including the compilation of the bibliography[1] and literature review (Biçer-Şimşir et al. 2009), the project initiated a testing program and developed a suite of protocols for both laboratory and field tests to provide reliable tools to enable informed decision making. The suite of tests developed was recently compiled into a manual including detailed test procedures, examples, and data collection sheets (Biçer-Şimşir and Rainer 2013).

This paper discusses the field tests designed for the comparison of injection grouts previously tested in the laboratory, and for quality control of materials and grout mixtures. These simple field tests have been designed for use by conservators and do not require specialized instrumentation or laboratory setups. Field tests carried out at the archeological site of Herculaneum provided critical feedback on the feasibility of these test methods on site in the context of collaborative research being carried out by the GCI and the Herculaneum Conservation Project (HCP). This research included the investigation of grouting methods and materials to overcome reported difficulties with grouting at the site, including injectability and lack of durability of grout injected into fine debris-filled and/or salt-laden walls.

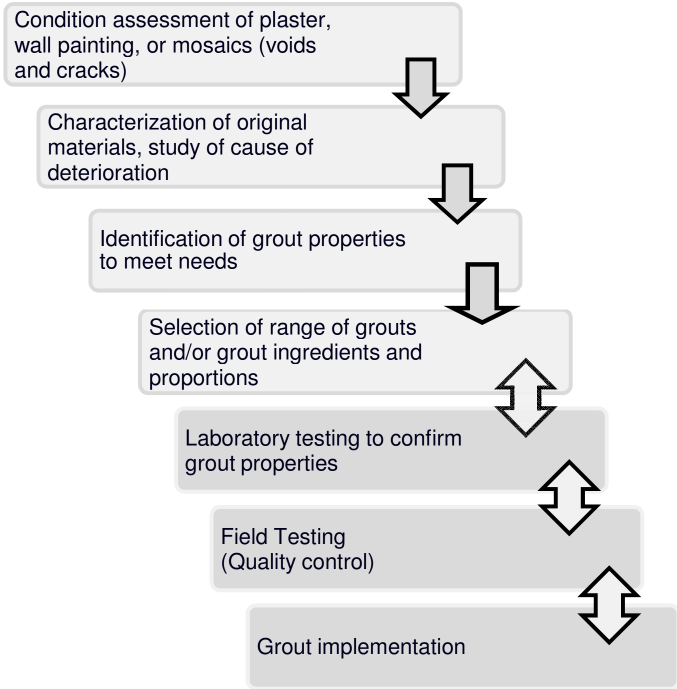


Figure 1
Grout selection and evaluation methodology

## TESTING PROGRAM

### Grout formulations and preparation

Four commercial and one custom-mixed grout with four modified versions were selected for field testing. Constituents and mix proportions of commercial grouts (Bresciani Malta 6002, Ledan TB1-ICR, LEIT, and PLM I) are given in Table 1. The specified amount of water recommended by the manufacturer was used to prepare these grouts.

The custom-mixed grout was based on the formulation for ICCROM grout (Ferragni et al. 1984) and contained 100 parts Lafarge Chaux Blanche NHL 3.5-z, 100 parts pozzolana *superventilata grigia,* 1 part sodium gluconate (10% solution by volume), 10 parts Primal B60A, and 125 parts of water, all by volume.[2] ICCROM grout volume proportions were converted into weight ratios both for the dry ingredients and water content (Table 2).

Table 1
Commercial grout mix proportions

| Grout name | Ingredients | Grout (g) | Water (g) |
|---|---|---|---|
| Bresciani Malta 6002 | hydraulic binder, silica powder, fluidizer, retaining and air-entraining admixtures | 100 | 94 |
| Ledan TB1-ICR | natural lime and hydraulic binders, inert silica, slate, pozzolan, fluidizer, retaining air-entraining admixtures | 100 | 79 |
| LEIT | hydraulic binders, silica aggregates, slate, pozzolan, fluidizer, retaining and air-entraining admixtures | 100 | 90 |
| PLM I | natural lime and hydraulic binder, inert materials and admixtures | 100 | 66 |

Table 2
ICCROM grout mix proportions

| Grout name | Lafarge Chaux Blanche NHL3.5-z (g) | St Astier NHL 2 (g) | Pozzolana grigia (g) | Primal B60A (cc) | Primal SF-016ER (cc) | Sodium gluconate solution (cc) | Water (g) |
|---|---|---|---|---|---|---|---|
| ICCROM | 90 | - | 92 | 10 | - | 1 | 125 |
| ICCROM_ w | 90 | - | 92 | 10 | - | 1 | 150 |
| ICCROM_w_NHL2 | - | 90 | 92 | 10 | - | 1 | 150 |
| ICCROM_SF | 90 | - | 92 | - | 10 | 1 | 125 |

Modified versions of the ICCROM grout were tested to evaluate the ability of suggested field tests to determine the effect of water content, binder, and additive changes in the formulation on related grout properties. ICCROM_w grout was prepared by increasing the water content of ICCROM grout by 20%; ICCROM_w_NHL2 grout was prepared by using St Astier NHL2 in place of Lafarge NHL3.5-z in ICCROM_w; and ICCROM_SF includes Primal SF-016 ER, a pure acrylic emulsion used in paint formulations, sent in place of Primal B60A or AC33 to Herculaneum by a local supplier.

High-speed mixing is necessary for injection grouts to disperse the fine particles and to obtain homogenized suspensions (Zajadacz et al. 2006). Therefore, a high-speed mixer with adjustable speed similar to those used in the laboratory was used in the field.

Grouts, with two exceptions, were mixed using a Caframo BDC 3030 stirrer with a maximum speed of 2800 rpm. Pre-measured distilled water (water and admixtures for ICCROM grout) was poured into the mixing bowl. All dry constituents (pre-mixed NHL and pozzolana for ICCROM) of the grouts were added to the water within 30 seconds while mixing at 200 rpm. The speed was then increased to 2000 rpm for the remainder of the 5-minute mixing time for all the grouts except LEIT, whose mixing speed was increased to 700 rpm and ICCROM grouts to 1000 rpm. After mixing, the fresh grout was passed through a 1-mm sieve to remove clumps.

PLM I_hm and ICCROM_hm grouts were mixed by hand for 5 minutes instead of high-speed mixing. Pre-measured amounts of water (water and admixtures for ICCROM) were added into dry ingredients, mixed for 5 minutes and were not sieved.

### Field test methods

Field tests described in this paper aim to quickly compare the properties of grouts in the wet state, and during curing and setting, without special instruments and setups. Field tests developed to evaluate performance properties including capillary water absorption, water vapor permeability, and bond strength are not discussed due to space constraints. A short description of each field test is included. Detailed procedures are provided in Biçer-Şimşir and Rainer (2013).

#### Injectability with syringe

The ability of a grout to fill a capillary network of granular materials under pressure is tested by pouring 20 mL of grout into a vertically held 60 mL syringe partially filled with 20 mL granular material (Figure 2), and applying pressure on the grout using the syringe plunger (Figure 3).

Figure 2
Syringes filled with crushed travertine

Figure 3
Injectability with syringe test of ICCROM_w into prewetted crushed travertine, classified as easy

Figure 4
Flow with syringe test of Bresciani Malta 6002 into dry crushed travertine, classified as difficult

Syringes are filled with representative building materials including crushed brick, travertine, and mortar (Ferragni et al. 1984, Biçer-Şimşir and Rainer 2013), and tested dry and prewetted. The grain size of the crushed brick and travertine is between 2–4 mm representing an approximately 0.32–0.64-mm crack width. Mortar used was debris removed from the location where the injection grouting was conducted. Prewetting is carried out pouring 100 mL distilled water into a vertically held syringe, partially filled with granular material, and letting the excess water drain for 5 minutes before injecting the grout. The injectability of the grout is classified as: easy (E) – if grout flows through the granular material and out of the syringe tip when pressure is applied; feasible (F) – if grout flows through the granular material and reaches the tip but does not flow through; or difficult ($D_L$) – if grout stops in the granular material before reaching the tip. The penetration distance, measured from the top of the granular material to the level the grout has reached, is recorded as L in millimeters.

### Flow with syringe

The ability of a grout to fill a capillary network of granular materials under gravitational force is tested by pouring 20 mL of grout into a vertically held 60 mL syringe that is partially filled with 20 mL granular material and tested dry and prewetted (Figure 4). The flow of the grout is classified as: easy (E) – if grout flows through the granular material and the tip of the syringe in 5 minutes or less; feasible (F) – if grout reaches the tip but does not flow through it in 5 minutes; difficult ($D_L$) – if grout halts in the granular material before reaching the tip after 5 minutes. The penetration distance, measured from the top of the granular material to the level the grout has reached, is recorded as L in millimeters.

### Expansion and bleeding

Eighty mL of grout is placed in a 100 mL graduated cylinder, and the amount of final bleeding and combined expansion are determined from the volume of accumulated water at the top of the freshly mixed grout with respect to the initial volume of grout and from the volume difference between the grout portion of the specimen and initial volume with respect to the initial volume.

### Wet density

A 5-mL syringe is filled with grout and weighed after air bubbles are removed. Wet density is calculated by dividing the weight of grout by the volume of grout (5 mL).

### Drying shrinkage

Dimensional change, including cracking, is determined by injecting 20 mL grout using a syringe without cannula in a plastic cup or “mortar cup” previously prepared with the mortar used for repairs on site.

The mortar cup is prepared by filling a container with mortar to the edge, pressing one end of a 45-mm-diameter plastic tube approximately 15-mm deep into the mortar, rotating it to hollow out a cylindrical cavity in the mortar, and finally removing the plastic tube and excess mortar from inside the cavity with a spatula to create a flat bottom (Figure 5).

The drying shrinkage of the grout is classified as: no shrinkage (NS) – no visible separation between grout and mortar and no visible cracking in the grout; medium shrinkage (MS) – a separation of less than 0.5 mm and/or a maximum crack size of less than 0.5 mm; or high shrinkage (HS) – a separation of more than 0.5 mm and/or a maximum crack size of more than 0.5 mm.

### Final setting time

The time of final setting is determined by periodically inserting a #12 metal cannula attached to a 60-mL syringe filled with granular material, weighing 100 g in total, into a plastic container or mortar cup filled with 20 mL grout (Figure 6). The syringe is held vertically and the tip of the cannula is positioned as close as possible to the surface of the grout without touching. The syringe is released and guided to fall directly into the grout every hour at 10 mm apart. Final setting time is the time when the cannula no longer penetrates into the grout.

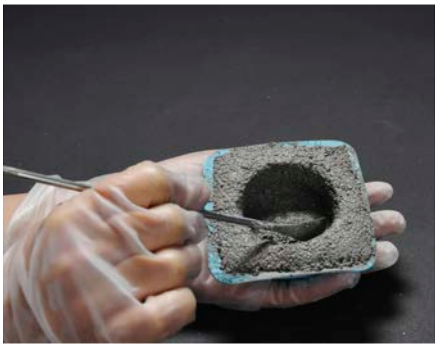

Figure 5
Preparation of a mortar cup, showing extra mortar removed by a spatula to create a cup

Figure 6
Final setting time test

## RESULTS AND DISCUSSION

### Injectability and flow

Results of the injectability with syringe test (Table 3) showed that additional water added to ICCROM_w grout improved its injectability both into dry crushed brick, from difficult to easy, and into dry crushed travertine, from feasible to easy. The results also demonstrated that prewetting of injection media with distilled water improved injectability for ICCROM grout and its modified versions, but it did not improve the injectability of PLM I into mortar. Injectability of PLM I into crushed mortar was more difficult than the other two tested media, demonstrating the importance of testing grouts using the injection media existing on site. The effect of prewetting

Table 3
Injectability with syringe results

| Grout name | Travertine | | Brick | | Mortar | |
|---|---|---|---|---|---|---|
| | dry | wet | dry | wet | dry | wet |
| ICCROM | F | E | $D_{10}$ | E | | |
| ICCROM_w | E | E | E | E | | |
| ICCROM_w_NHL 2 | | | $D_{15}$ | E | | |
| ICCROM_SF | F | E | $D_{12}$ | E | | |
| ICCROM_hm | | | $D_{8}$ | F | | |
| PLM I | E | E | E | E | $D_{10}$ | $D_{10}$ |
| PLM I_hm | E | E | E | E | | |
| LEIT | E | E | E | E | | |
| Ledan TB1-ICR | E | E | E | E | | |
| Bresciani Malta 6002 | E | E | E | E | | |

| | |
|---|---|
| E | Easy |
| F | Feasible |
| $D_{L}$ | Difficult |

on injectability and flow could not be determined for commercial grouts since their injectability in dry media was already easy. Injectability of ICCROM_w grout into dry crushed brick went from easy to difficult when NHL 3.5 was replaced with NHL 2. Injectability of ICCROM grout did not change noticeably when Primal SF-016 ER was added in place of Primal B60A. Finally, results showed that hand mixing reduced the injectability of ICCROM grout (ICCROM_hm) into crushed brick, but did not affect the injectability of PLM I (PLM I_hm).

Comparison of injectability (Table 3) and flow with syringe (Table 4) test results showed that penetration of grouts under pressure and gravity are different. Therefore, the flow with syringe test is necessary for gravity-based injection grouting (e.g., floor mosaics). All grouts tested under gravity, except LEIT for all conditions and Ledan TB1-ICR for prewetted cases, demonstrated reduced ability to penetrate into granular materials. While the extra water in ICCROM_w clearly increased injectability, and the use of NHL 2 (ICCROM_w_NHL2) and hand mixing (ICCROM_hm) reduced injectability under pressure, the addition of water led to a much smaller increase and the use of NHL 2 led to a much smaller decrease of the flow through granular material (Table 4). Hand mixing of ICCROM did not affect the penetration under gravity, but hand mixing of PLM I reduced the penetration, especially into the prewetted media.

Injectability and flow with syringe tests also provided information on the ability of a grout to retain its water when subjected to suction created by adjacent porous materials. The higher the water retention of a grout, the longer the distance it is expected to move without losing its fluidity. Results showed that ICCROM grout and its modified versions lost their water quicker than the commercial grouts.

### Expansion and bleeding

ICCROM_w and LEIT grouts had the highest final bleeding (1%) among all grouts tested (Table 5). Grouts that are well formulated, properly proportioned

Table 4
Flow with syringe results

| Grout name | Travertine | | Brick | |
|---|---|---|---|---|
| | dry | wet | dry | wet |
| ICCROM | $D_4$ | $D_{19}$ | $D_1$ | $D_9$ |
| ICCROM_w | $D_9$ | $D_{22}$ | $D_6$ | $D_{15}$ |
| ICCROM_w_NHL 2 | | | $D_2$ | $D_6$ |
| ICCROM_hm | | | $D_2$ | $D_6$ |
| PLM I | $D_{22}$ | F | $D_8$ | F |
| PLM I_hm | $D_{19}$ | $D_{29}$ | $D_9$ | $D_{35}$ |
| LEIT | E | E | E | E |
| Ledan TB1-ICR | F | E | $D_{21}$ | E |
| Bresciani Malta 6002 | $D_8$ | $D_{13}$ | $D_6$ | $D_{11}$ |

| | |
|---|---|
| E | Easy |
| F | Feasible |
| $D_L$ | Difficult |

and mixed should not segregate or bleed excessively. Separation of liquid and solids negatively affects performance characteristics including drying shrinkage, intrinsic and bond strength. Segregation also causes clogging during injection.

Only Bresciani Malta 6002 demonstrated a combined expansion of 1%, which may require special attention. Similar to the reduction of volume after setting, increase of volume may cause issues with thin, fragile plasters. Malta 6002 is marketed as a lightweight grout, and expansion results indicate that the grout may include admixtures that create air bubbles and increase volume.

### Wet density

The wet density values obtained for the tested grouts were between 1.25–1.55 g.cm$^{-3}$ (Table 5). Bresciani Malta 6002, marketed as a lightweight grout, had the lowest wet density (1.25 g.cm$^{-3}$), followed by Ledan TB1-ICR (1.31 g.cm$^{-3}$). Additionally, field testing indicated that wet density measurement using a syringe was sensitive enough to determine density changes due to addition of water, from 1.50 g.cm$^{-3}$ to 1.45 g.cm$^{-3}$, and the use of NHL 2 instead of NHL 3.5, from 1.45 g.cm$^{-3}$ to 1.43 g.cm$^{-3}$.

Wet density is an important parameter if added weight as a result of grouting might cause failure of the architectural surfaces, such as grouting of large voids in ceilings and vaults. Since grout is heavier when fresh, and the surrounding materials have lower strength when wet, it is important to calculate added weight during grouting.

### Drying shrinkage

Results in Table 5 showed that extra mixing water in ICCROM_w increased the separation between the mortar cup and grout from 0.45 mm to 0.65 mm. Relatively quick absorption of additional water by the substrate was also visible as a darker shaded area around the cup filled with ICCROM_w (Figure 7). The use of NHL 2 further increased the separation to 0.95 mm (Figure 8). Drying shrinkage of ICCROM_SF grout in mortar cup (1.50-mm

**Figure 7**
Drying shrinkage test. ICCROM (left), ICCROM_w and ICCROM_w_NHL 2 (right) filled mortar cups

**Figure 8**
Simple adhesion test using drying shrinkage specimens. Grout removed easily with minimal residual in the cup – low adhesion

**Table 5**
Combined field test results

| | Expansion and bleeding | | Wet density | Drying shrinkage in mortar cup | | | Final setting time | |
|---|---|---|---|---|---|---|---|---|
| Grout name | Combined expansion (%) | Final bleeding (%) | (g.cm$^{-3}$) | Separation size (mm) | Crack size (mm) | Class | mortar cup (hr) | plastic cup (hr) |
| ICCROM | 0 | 0 | 1.50 | 0.45 | 0.00 | MS | 2 | 9 |
| ICCROM_w | 0 | 1 | 1.45 | 0.65 | 0.40 | HS | 3 | 17 |
| ICCROM_w_NHL 2 | 0 | 0 | 1.43 | 0.95 | 0.00 | HS | 16 | |
| ICCROM_SF | - | - | - | 1.50 | 0.85 | HS | - | - |
| ICCROM_hm | 0 | 0 | 1.50 | - | - | - | - | 9 |
| PLM I | 0 | 0 | 1.55 | 0.25 | <0.10 | MS | 11 | 13 |
| PLM I_hm | 0 | 0 | - | 0.25 | <0.10 | MS | 11 | - |
| LEIT | 0 | 1 | 1.49 | 0.10 | 0.10 | MS | 13 | 40 |
| Ledan TB1-ICR | 0 | 0 | 1.31 | 0.30 | 0.10 | MS | 11 | 22 |
| Bresciani Malta 6002 | 1 | 0 | 1.25 | 0.10 | 0.10 | MS | 13 | 13 |

separation) was distinctly different than the drying shrinkage of ICCROM grout. This observation pointed out that Primal SF-016 ER, sent as a substitute of Primal AC33 or B60A, was not appropriate for grout applications.

In general, commercial grouts, which retained their water longer against the suction of substrate, demonstrated smaller cracks and separation. However, laboratory tests showed that LEIT had the highest volumetric drying shrinkage of all grouts tested, and this was not reflected in the field test results. The main reason for this was the way in which the measurements were taken. The surface of the LEIT grout in the mortar cup was not leveled and had gradually sunk almost 2 mm at the center when compared with the edges, indicating significant drying shrinkage but minimal cracking.

Drying shrinkage results demonstrated that decreased volumetric stability generally leads to the formation of cracks, which in turn may cause loss of the bond between the grout and substrate, and loss of grout strength. A simple way of assessing the adhesion between the grout and the mortar cup in the field is to attempt to pop out the grout from the mortar cup after curing using a small spatula. If the grout pops out easily and the grout residual in the cup is minimal (Figure 8), it is classified as low adhesion. If the grout pops out with some grout adhered in the cup without damaging the cup, it is classified as adequate adhesion.

### Final setting time

Additional water in ICCROM_w delayed the final setting time for one hour in a mortar cup (Table 5). The use of NHL 2 instead of NHL 3.5 showed a clear increase in the time of final setting from 3 to 16 hours. The time measured depends on the type of container used; generally, it is longer for a grout in a plastic container (Table 5). The range of final setting times of commercial grouts was between 10 and 13 hours in mortar cup and between 13 and 40 hours in plastic cup.

The time of setting, or onset of rigidity, is of interest particularly in situations where a more rapid set may be desired (e.g., fragile plasters, vaults, large voids), or cases where it is necessary to know when supports can be removed. This test is also useful to control the quality of the hydraulic binders in the field. Hydraulic binders such as natural hydraulic lime will lose their binding ability and demonstrate setting delays or no setting when prematurely exposed to moisture.

## CONCLUSION

Tests provided in this paper are simple, relatively quick, easily carried out on site, and can show: a) intended (e.g., to improve injectability and flow) or unintended water content modifications of the grout and effects on the volumetric stability of the grout when it is fresh (bleeding) and after setting (drying shrinkage); b) ingredient changes due to a new shipment or inappropriate storage conditions (e.g., binder or admixture); and c) mixing and preparation changes.

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. They can help overcome issues of grout performance by providing information on the origin of the problem, and conditions at the site that may have led to the performance issue (e.g., environmental conditions, materials, preparation, etc.), and by enabling identification of modifications that may help solve the problems (increase injectability, reduce shrinkage, etc.).

Although field tests can be very useful, 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 laboratory testing. It is important to know that the results obtained may have a high coefficient of variation and are rarely reproducible, mainly due to changes in environmental conditions and varying substrate properties.

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

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## NOTES

1 www.getty.edu/conservation/our_projects/field_projects/grouts/grouts_bib_2_17_09.pdf (accessed 31 January 2014).

2 In this study, Primal AC33, which has been discontinued, was substituted by Primal B60A with a similar composition (Torraca 2006).

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## MATERIALS LIST

Bresciani Malta 6002 (hydraulic grout)
www.brescianisrl.it

Lafarge Chaux Blanche NHL3.5-z (natural hydraulic lime)
www.ctseurope.com

Ledan TB1-ICR (hydraulic grout)
www.tecnoediletoscana.it

LEIT (hydraulic grout)
www.phaseitalia.it

PLM I (hydraulic grout)
www.ctseurope.com

Primal B60A (water-based acrylic emulsion containing 46–47% solids of a copolymer ethyl acrylate and methyl methacrylate)
Rohm & Haas
www.imaronline.com

Primal SF-016 ER (pure acrylic emulsion containing 50–51% solids)
www.imaronline.com

Sodium gluconate
www.sigmaaldrich.com

St Astier NHL 2 (natural hydraulic lime)
www.limes.us/contact

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