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Characterization of Historic Mortar
Old Parish House
College Park, MD

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Characterization of Historic Mortar

Introduction
As a prelude to many restoration projects and maintenance campaigns, materials testing and analysis is often considered, the objective being to provide information that will be useful in understanding the history of a building and also to provide insight into materials which could be appropriate for future maintenance campaigns. One test which is regularly undertaken is "mortar analysis," wherein the composition of a composite material, such as mortar, stucco or plaster, is assessed. Although the sophistication of different analytical methods varies, test protocols generally seek to identify whether materials such as cement, lime and sand are present, and in what quantities.

This analysis focuses on the composition of one mortar from Old Parish House in College Park, MD. This sample was procured by a conservator from deGruchy Masonry as part of a masonry consultation, transmitted to Keystone Preservation, and catalogued as follows:

- Brick bedding mortar

Information included in the material transmittal indicated that the structure from which this sample was taken was built in 1817. The sample was noted as having been removed from an exterior brick joint with dry exposure.

The test protocol employed during this analysis is capable of distinguishing characteristics which are typically associated with binders such as lime or cement. However, it is important to note that analysis is subjective rather than unequivocal – based on an interpretation of data and an assessment of physical properties. The level of detail offered by this protocol is typically sufficient to establish basic information about composition. Interpretation relies not only on data produced during testing, but also on physical characteristics of the material, such as color, texture, hardness and cohesiveness.

The objective of this analysis was to characterize the composition of historic materials and to provide recommendations for close color simulations within LimeWorks.us line of Natural Hydraulic Lime-based Ecologic Lime Mortars and St. Astier's Natural Hydraulic Lime products. Natural Hydraulic Lime (NHL) is a traditional building material which offers certain advantages over non-hydraulic lime materials, lime-cement hybrids and cement-based materials. Whereas materials based on slaked lime putty or dolomitic lime cure with a process of carbonation over extended periods of time, NHL achieves a cure time more quickly. Additionally, materials based on NHL are typically more durable than those based on non-hydraulic limes, yet more flexible and more vapor-permeable than lime-cement hybrids or cementitious materials. This report discusses composition only, as opposed to replication. Upon request, LimeWorks.us will provide a stock material or custom simulation to match the color and texture of the historic materials.

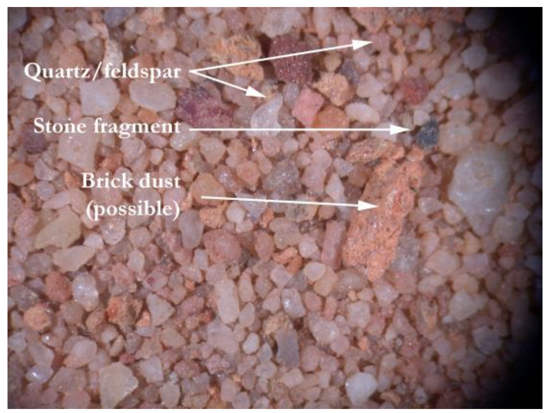
Analytical Summary

Brick bedding mortar

This mortar displays evidence of physical and reactive properties that suggests a mix based on a combination of lime, sand, clay and brick dust.

- Reactivity: Reactivity: moderately vigorous, voluminous production of carbon dioxide when reacted with hydrochloric acid. The quantity of carbon dioxide was substantial (a characteristic which is commonly associated with traditional lime mortars).
- Hardness: Moderate, cohesive. In general terms, lime-based materials are typically softer and easier to break than cementitious materials - the latter of which sometimes require significant impact from a mortar and pestle or even a hammer to break apart. Although it is cohesive, dry and stable, this mortar required only minimal impact to crush, and minimal impact to further pulverize – consistent with what would be expected from a clay-bearing lime mortar.
- Color: *pink* (Munsell 7.5YR 8/4). Whereas cement-bearing materials are often gray, those based on lime often tend to be white or buff in color – or light brown to pink if they contain clay (color varying in accordance with the clay source). The *pink* color of this mortar can be attributed to the presence of lime (white), sand (light reddish brown), clay (light brown to red) and possibly brick dust (light red).

Photomicrograph of core of brick bedding mortar (incident color-balanced light @ 10x magnification). White binding material attached at either side of this fragment does not appear to be part of the matrix.



Photomicrograph of aggregate extracted from brick bedding mortar (incident color-balanced light @ 10x magnification).

The reactive and physical characteristics of this mortar suggest that it is contains a lime binder. It is not known whether the lime is high calcium or dolomitic, or whether any hydraulic components are present. Additional testing including instrumental analysis (X-Ray Diffraction, SEM-XEDS and other methods would be required if additional clarification on binder is required). Characteristics are consistent with what might be expected from a mix based on 2 parts binder (lime and clay) and 5 parts aggregate (sand and possibly brick dust).

LimeWorks.us will issue a recommendation for the use of an NHL-based material to closely match the color and texture of the mortar.

Methodology

Compositional evaluation for this material was completed by Keystone Preservation Group, Inc. under a cooperative relationship with LimeWorks.us. Within this relationship, LimeWorks.us typically provides samples of the historic material, a description of the building (if applicable) and observations which were made when the material was removed (if applicable). Keystone Preservation Group completes analytical testing and provides LimeWorks.us with data on the historic material and also advises whether stock (or blended) materials within LimeWorks.us' Ecologic Lime Mortar line of materials simulate significant visual characteristics such as color and texture. In some instances, if a standard material does not simulate color and texture, LimeWorks.us may produce a custom material for specific individual projects. Keystone Preservation Group warrants the analytical information contained herein to the greatest degree possible. Issues such as material appropriateness, installation, and performance are the responsibility of the owner's masonry restoration consultant and/or the installer.

Each specimen was examined according to properties of color, texture, hardness, homogeneity, stability and relative porosity. Samples were examined visually and microscopically with a Nikon SMZ-2T trinocular reflected light microscope.

The approximate composition of the material was determined with a calcimeter conforming to the parameters of the Jedrzejewska analytical method.[1] This technique essentially breaks down a sample into constituent parts and provides data on the nature of the binder by gauging the extent of its reaction with hydrochloric acid (HCl). As HCl dissolves bicarbonates of calcium carbonate-based ($CaCO_3$) compounds found in lime and (to a lesser extent) cement binders, carbon dioxide (CO_2) is produced.[2] Data obtained during experimentation was compared with published experimental standards based on known mixes to arrive at conclusions about the composition of all samples.[3] Aggregate which forms the insoluble portion of the material was isolated, retained and washed, while extremely fine particulates were separated from solution, filtered and retained. The aggregate was dried and weighed, evaluated according to particulate size with a Standard U.S. Sieve Series Tyler Equivalent Sieve Stack, and examined microscopically for particle shape, color, opacity and mineralogy. Fine particulates, once filtered, were dried, weighed, and examined visually and microscopically.

The degree of testing discussed herein is sufficient to establish a basic understanding about the composition of the materials supplied to our laboratory. That said, gravimetric analysis and tests which utilize acid digestion constitute an inexact science, relying substantially on the experience and interpretation of the analyst as well as comparison with materials with known composition. As such, this report should not be interpreted as providing unequivocal compositional data on the material(s) which have been transmitted to our laboratory. Petrographic analysis including examination of thin sections in transmitted polarizing light and/or elemental analysis would be required to identify mineral phases which are specific to different types of cementing material and to unequivocally quantify the amount of lime and/or cement present. If analysis in accordance with testing procedures described in ASTM C1324 is desired, microchemical characterization may be expanded

[1] Hanna Jedrzejewska, "Old Mortars in Poland: A New Method of Investigation," Studies in Conservation, Volume 5, Number 4, 132-138.

[2] Calcimeter analysis provides information beyond standard gravimetric/acid digestion. Although not unequivocal and subject to interpretation, it provides data on acid-soluble portions which do not evolve carbon dioxide when reacted with HCl. This is especially important to the analysis of cementitious mortars that naturally contain complex soluble silicates which dissolve in acid but do not evolve carbon dioxide. Limitations to this analytical method include the lack of published standards on common masonry mixes and mixes which incorporate natural and artificial pigments.

[3] John Stewart and James Moore, "Chemical Techniques of Historic Mortar Analysis," Bulletin of the Association for Preservation Technology, Volume 14, Number 1 (Washington: Association for Preservation Technology, 1982), 11-16.

upon with elemental analysis using techniques such as X-Ray Diffraction (XRD), petrography and/or physical characterization of thin sections using transmitted and polarized light microscopy.

Compositional Characteristics

Binders

Analyzing the nature of the binder provides information valuable in determining the composition of the original material. Binders are generally composed of calcium-carbonate based materials with impurities and additives incorporated to affect physical properties such as cure, hardness, color and durability. The data below represents the percentage weight within the sample that may be attributed to calcium carbonate, complex soluble silicates (solubles, including soluble material from cements and pigments) and aggregate.

Standards referenced below[4] have been culled from previously-completed research and experimentation to provide benchmark data regarding compositional percentages for standard mixes. Although imperfect, experimental data below exists for compositional proportions based on weight only; conclusions are based on the mathematical conversion of weight percentages to volumetric mixes.

	Mix	**$CaCO_3$**	**Solubles**	**Sand**
1:3	lime:sand (SM).[5]	10.0 ± 0.4%	2.4 ± 0.7%	87.6 ± 0.3%
1:3	lime:calcareous sand (SM).[6]	26.2 ± 1.3%	3.0 ± 0.9%	70.9 ± 2.2%
1:3	lime:clayey sand (SM)	10.2 ± 0.2%	13.2 ± 0.6%	76.6 ± 0.9%
2:5	hydraulic lime:clayey sand (JCF).[7]	8.78± 0.06%	17.69 ± 3.01%	73.54 ± 2.95%
2:5	dolomitic lime:clayey sand (JCF).[7]	6.57± 0.25%	10.10 ± 0.63%	83.33 ± 0.98%
2:5	high calcium lime putty: clayey sand (JCF)[7]	13.49 ± 1.27%	5.78 ± 1.02%	80.72 ± 2.29%
1:1:5	Portland cement: high calcium lime:clayey sand (JCF).[7]	10.60 ± 0.87%	21.76 ± 5.99%	67.65 ± 6.87%
2:1:5	lime:pozzolanic cement:sand (SM)	11.0 ± 0.1%	6.6 ± 0.4%	82.4 ± 0.3%
100%	Portland cement (SM)	7.2 ± 0.4%	92.1 ± 0.3%	0.6 ± 0.2%
1:3	Portland cement:clayey sand (SM)	7.9 ± 0.1%	29.6 ± 1.6%	62.6 ± 1.6%
1:3	Portland cement:sand (SM)	6.4 ± 0.4%	27.4 ± 3.0%	66.2 ± 3.4%
1:3	Roman cement: sand (SM)	8.5 ± 0.6%	16.1 ± 0.2%	75.3 ± 0.4%

[4] In the chart, "SM" refers to standards published in Stewart and Moore's article and "JCF" refers to data from Frey's thesis.

[5] Laboratory analyses indicate that the process of carbonation, by which wet lime converts to solid calcium carbonate, is indefinite. Therefore, lime which has aged for an extended period of time will evolve more carbon dioxide.

[6] Total calcium carbonate content includes soluble bicarbonates from calcareous (calcium-based) aggregate such as crushed limestone or marble dust.

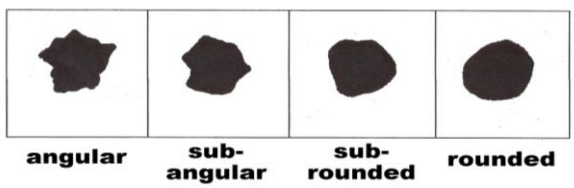
[7] Analysis performed on replication mix evaluated (five years after cure) in J. Christopher Frey *Exterior Stuccoes as an Interpretive and Conservation Asset: The Aiken-Rhett House, Charleston, SC* (Masters' Thesis in Historic Preservation, University of Pennsylvania, 1997). Please note that data is provided for comparative purposes only; actual volumetric percentages will vary based on types of lime, types of sand, mix consistency, etc.

Aggregate
Because sand is so important in determining not only how a material performs but also in producing its color and texture, a careful examination of the aggregate was conducted. Laboratory examination included visual analysis and an evaluation of particle size.

All sands were passed through a U.S. Standard Sieve Series Sieve Stack to evaluate the average distribution.[8] The sieve screens used in this analysis possess the following dimensional equivalents:

Screen #	Tyler equivalent mesh	Opening (inches)	Opening (metric)
8	8	.0937	2.36 mm
16	14	.0469	1.18 mm
30	28	.0234	600 μ
50	48	.0117	300 μ
100	100	.0059	150μ
200	200	.0029	75μ

Materials with rough surface textures tend to be based on sands whose grains are retained on the lower-numbered screens. Smoother materials are often based on finer particulates. Well-graded materials are characterized by relatively even distribution of particulates across all screens. Data reported below provides a comparative analysis of particulate grain sizes for the materials analyzed.



Typical grain shapes within sand components.

[8] Sieves are graded in a series so that particles progressively pass through a series of screens (with screen #8 representing the largest mesh and screen #200 the smallest) until they are retained. The amount of material retained on each screen is recorded and then compared with the amounts retained on other screens to provide numerical averages for different particulate sizes.

Analytical Datasheet

Brick bedding mortar

The physical properties and reactive characteristics of this material are consistent with what would be expected from a mix based on lime and sand with clay and possibly brick dust.

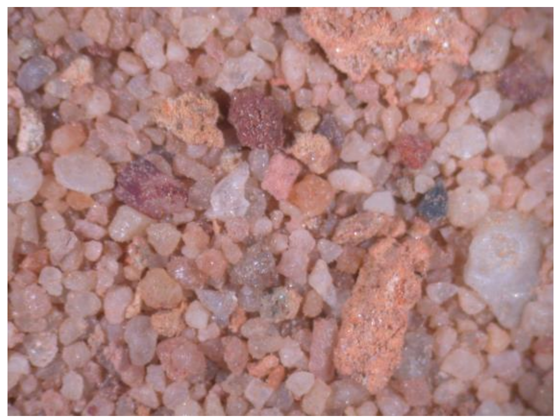
Analytical Method

Calcimeter	✓
ASTM C1324	
Basic/acid digestion	✓

Binder

$CaCO_3$	~ 12.41%
Solubles	~ 18.29%
Sand	~ 69.29%

Photomicrograph of core of brick bedding mortar – based on lime and sand with clay and possibly brick dust(incident color-balanced light @ 10x magnification).



Photomicrograph of aggregate extracted from brick bedding mortar - sand based on quartz/feldspar with stone fragments and possibly brick dust (incident color-balanced light @ 10x magnification).

Reacting this material with hydrochloric acid (HCl) produced results that suggest the presence of a binder which is based on lime. The production of a moderately vigorous, voluminous evolution of carbon dioxide (CO_2) suggests an abundance of carbonate (CO_3), which is a chief component in lime (calcium carbonate or calcium, magnesium carbonate $CaCO_3$ or Ca, $MgCO_3$). Data also suggests the presence of a moderate quantity of non-evolving, acid soluble compounds – likely attributable to clays that were bound into or added to the original sand source, and also possible brick dust (some of which can dissolve in acid). Instrumental analysis and petrography would be required to confirm compositional quantities as well as to identify specific compounds and mineral phases which might be associated with specific types of lime (e.g. high calcium, dolomitic, hydraulic).

Physical Properties

The color of this mortar is *pink* (Munsell 7.5YR 8/4) – a shade which can be attributed to the presence of lime (white), sand (light reddish brown), clay (light brown to red), and possibly brick dust (light red). White binding material is attached to the edges of certain parts of the sample, but does not appear to be part of the matrix. Although it is cohesive, stable and dry, this mortar requires minimal impact from a mortar and pestle to break into fragments, and minimal pressure to further pulverize.

Aggregate
This is a *light reddish brown* aggregate which is based primarily on quartz/feldspar with stone fragments and possibly brick dust. Clarity is transparent to translucent. Grain shape is primarily rounded to subrounded. In general terms, this sand is more fine than coarse, with almmost 85% of all grains retained on or below the #50 sieve screen screens (see Table and images below).

Extracted sands were recorded by weight using the sieve gradation analysis and converted to percentages of the sample. The table below documents quantitative results from sieve gradation analysis of the extracted sands from the historic mortar.

Screen Size	#8	#16	#30	#50	#100	#200	Fines
Sands (retained)	1.47	2.06	10.03	65.48	18.58	2.06	0.29

The percentage of sand grains retained on each sieve screen are visually represented below. The grain size decreases from left to right. Of note is the fact that fines were found to contain a combination of fine sand, fine brick dust and/or fine clay; it has not been possible either to isolate and quantify each individual component, or to interpret how each affects the mortar on a quantifiable level.