

Mortar Analysis Part 2: Analytical Methods

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Introduction

Methods for mortar analysis can vary depending upon the objectives for the work. Information sought through analysis of historic mortars includes the following:

- the composition of the mortar, including the original binder components; the mineralogy, character, and source of the aggregate; and the presence and type of admixtures
- the proportions of the original mix, specifically binder-to-aggregate ratio, but possibly also the original water content
- the physical properties of the mortar, including primarily porosity but also absorption and permeability
- the mechanical properties, or strength, of the mortar

Though all four characteristics must be evaluated to fully understand a historic material, mortar analysis is often equated with determination of just the first two. The focus of this article is therefore a discussion of analytical methods used for determining mortar composition and proportions.

Sampling

Before any analysis can be done, mortar samples must be collected. Obtaining the right number of the right kind of samples is critical to the outcome of the analysis. Unfortunately, selecting the right samples can be an extremely complicated process (Fig. 1). The importance of proper sampling will be addressed in detail in a subsequent Practice Point.

Selecting an Analytical Method

Theoretically, any method suited to the compositional analysis of inorganic materials could be applied to mortar. Selecting the right method requires a clear understanding of how available methods work and what type of result they return. For example, does the method identify compounds (i.e., calcium silicate hydrate) or elements (i.e., calcium and silicon)? Can it be used to identify both crystalline and amorphous compounds or only compounds that have a crystalline structure? Does it allow for distinguishing two compounds that



Fig. 1. Detail of a mortar joint in an eighteenth-century masonry wall, showing three layers of mortar. Multiple layers of mortars of different compositions and ages complicate the process of taking samples for mortar analysis. Images by the author, unless otherwise noted.

are identical except for their bound water content? Is the method quantitative, returning a numerical content value; qualitative, returning only content information; or somewhere in between (semiquantitative)? What size sample is needed to perform the analysis? Does the sample have to be solid and intact, or can powdered material be used? What kind of sample preparation is required, and how might the sample preparation affect the results? What are the detection limits for the method — how much of a particular element or compound must be present before it can be identified? What materials in the sample might interfere with getting accurate results from the method?

An extensive knowledge of historic mortar materials and their reaction chemistries is also needed to choose the right analysis method. Based on the discussion of mortar materials in Practice Points Number 5, the composition of hardened historic mortars might seem straightforward.¹ A hardened mortar prepared from lime would consist primarily of calcium carbonate, and a hardened cement mortar primarily of calcium silicate hydrate. A hardened hydraulic-lime mortar would contain both calcium carbonate and calcium silicate hydrate.

In reality, the situation is not so simple. The binder composition of a hardened mortar varies not only with the character and quantity of the original components but also with the age of the mortar and the type and degree of exposure. For example, portland-cement



Fig. 2.

An example of tabby, a historic material prepared from lime, shells, sand, and water, which historically was covered with stucco. The aggregate in this case is readily distinguishable as acid-soluble shells. This image shows exposed tabby with a portion of the stucco still intact from Cannon's Point Plantation in Georgia. Courtesy of Mary Striegel.

mortar will contain calcium hydroxide in addition to calcium silicate hydrates; lime mortars may not be completely carbonated and may also contain hydrated calcium silicates and calcium hydroxide, depending upon the composition of the source rock from which the lime was prepared. Dolomitic limes yield mortars with calcium-magnesium carbonates. Hydraulic limes and natural-cement binders contain similar components that can be difficult to distinguish on the basis of chemistry alone. Hardened mortars also typically contain unaltered residues of the original binder materials, further complicating their chemistry.

Then there is the consideration of the aggregate, which, though often a silica sand, could also be crushed stone, shells, slag, or a number of other materials or combinations of materials (Fig. 2). Aggregates can be composed primarily of silicate minerals but could also contain minerals such as calcium carbonate in various forms, dolomite, and other minerals similar to those that might make up a hardened mortar binder. The small proportion of binder relative to the aggregate complicates interpretation of analytical results, in that the aggregate minerals can overwhelm the analytical results, making detection of the binder components more difficult.

Finally, transformations in the hardened mortar over time (particularly carbonation), the intimate intermingling of the binder with the finest components of the aggregate, and the extremely small crystal size of the minerals in the hardened binder further complicate selection of suitable analytical methods.

Analytical Methods Used in Practice

Because of the complexity of historic mortars, many different methods (and combinations of methods) have been used by researchers worldwide in their attempts to discern the original components of hardened mortars. An annotated bibliography of work from the mid- to late 1980s through the late 1990s is included in the work of Elizabeth Goins.² A more interesting type of review was done by Kara Dotter, who evaluated the frequency with which certain techniques were cited in the mortar-analysis literature in publications primarily from the 1990s through 2005.³ Though the list of papers reviewed by Dotter is representative rather than comprehensive, petrographic analysis of mortar thin sections using polarized-light microscopy was the most frequently used method. X-ray diffraction analysis (XRD) was the second most frequently used method, followed by wet chemical analysis (including acid-digestion analysis) and scanning electron microscopy combined with energy-dispersive spectroscopy (SEM/EDS). The utility of these particular methods was reiterated by the international commu-

nity in the State-of-the-Art Report of the International Union of Laboratories and Experts in Construction Materials, Systems, and Structures (RILEM) technical committee concerned with mortar analysis.⁴

The predominant use of these particular methods is based on the understanding of three significant factors: first, that mortar binders are made up of minerals and compounds with distinctive optical characteristics that are identifiable using the right optical techniques; second, that remnants of unaltered original mortar binder materials can often be found in hardened mortars, and these materials, too, are discernable using optical techniques; and finally, that compounds that cannot be discerned with optical techniques due to their small crystal size can be distinguished using XRD analysis and SEM/EDS. Each of these methods will be discussed in some detail.

Petrographic Analysis

The term *petrographic analysis* is used both broadly to include several different types of analysis and more narrowly to mean only polarized-light microscopy. At minimum the method involves examination of a sample at low magnification in reflected light with a stereomicroscope, followed by mounting and grinding the sample to prepare a thin section, which is then examined in transmitted light with a polarized-light microscope.

Many details of a mortar can be observed under low magnification with a stereomicroscope. For example, the proportions of aggregate, binder, and voids can be estimated. Entrained air voids can be distinguished from entrapped air, and the presence of drying shrinkage cracks noted. The composition and character of the aggregate can be evaluated; the general character of the binder can be observed; and binder-to-aggregate ratios can be estimated visually (Fig. 3).

Polarized-light microscopy (PLM) yields significantly more information than stereomicroscopic examination alone and has been used for many years in the study of concrete. The minerals in the aggregate are readily identifiable, and their characteristics reveal a great deal about the aggregate source. Relict binder grains (unhydrated cement, underburned or overburned lime, etc.) can be observed and identified (Fig. 4). Though the grain size of the binder minerals is typically too small for positive identification by optical methods, characteristics of certain binder components (such as carbonates and calcium silicate hydrates) are well enough known that binder composition can usually be determined to some degree. Charcoal, brick fragments, and lumps of lime putty or other binder materials, as well as pozzolanic additives, are easily distinguished (Fig. 5). Void characteristics can also be clearly distinguished. Additional compositional and textural information can be gathered through the use of special staining and impregnation techniques.⁵ An ex-

cellent review of PLM for use in evaluation of historic mortars was recently published.⁶

Quantitative analysis can be performed through point counting, a petrographic technique that allows for determination of volumetric proportions of aggregate, binder, and void space, as well as compositional characteristics (i.e., unhydrated cement grains, proportion of different aggregate minerals, etc.).⁷ A thick cross section of a mortar sample, abraded slightly to smooth the surface and viewed in reflected light, might be used for the same determinations (except binder characterization) without the expense of thin-section preparation. Modern computer image-analysis methods were developed to improve upon point-counting methods. Image analysis requires a microscope equipped for photomicrography (to capture the images) and specialized computer software (to analyze the images and generate data). Image-analysis techniques can also be used to quantify aggregate grain-size distribution and composition (Figs. 6 and 7). Image analysis is a particularly promising technique for quantitative evaluation of mortars, and work is underway to improve its utility for this purpose.⁸

When prepared properly, thin sections can also be used for other types of instrumental analysis. A polished thin section⁹ can be used in a scanning electron microscope (SEM), giving the opportunity for elemental analysis of the binder and aggregates. High-magnification imaging can be used in conjunction with elemental analysis to identify components of the binder based on crystal structure and chemistry (Fig. 8).

XRD and SEM/EDS

The principles and procedures of XRD and SEM/EDS were clearly described in Practice Points Number 4 and so will not be addressed in detail here.¹⁰ Essentially, XRD is a technique performed on powdered samples that allows for the identification of crystalline materials. Though typically considered a qualitative technique, the method can be used quantitatively through the application of certain algorithms and has been used in this manner to interpret the composition of mortar binders.

One difficulty with XRD analysis is that significant peaks for different minerals can overlap. Complementary use of PLM can usually mitigate this difficulty. An abundance of amorphous, or non-crystalline, materials can obscure smaller peaks in certain ranges of the scan, as can abundant iron-containing minerals, but again, PLM can be used to determine if these types of interferences are likely to be a problem.

SEM/EDS allows for visualization of a sample in three dimensions and has a much higher practical magnification than PLM. Both features are extremely useful for developing a better understanding of the binder in a hardened mortar. When utilized in BSE (backscattered electron) mode and/or combined with



Fig. 3. Stereomicroscopic view of two mortar samples, 10x. Both of these samples are lime mortars of similar character, but differences between the binder aggregate ratios and aggregate size are easy to see. At higher magnifications, distinctive characteristics of the binder and voids are clearly visible.

EDS, SEM can also yield useful information about the elemental composition of binder components. This information is particularly helpful for characterizing amorphous materials, which cannot be identified by XRD. Image-analysis techniques can also be applied to SEM/BSE images in the same way they are used for photomicrographs to determine overall proportions of binder, void, and aggregate.

Other Methods

There are other methods that have been applied to the analysis of mortars with varying degrees of success. Thermal analysis techniques, such as differential thermal analysis (DTA), differential scanning calorimetry (DSC), and thermogravimetric analysis (TG), all rely on detecting changes in an unknown material upon heating, compared to a material that does not change when heated. These changes include loss of bound water (or water of crystallization), carbonate, and other chemically bound components that occur at known temperatures for particular compounds. Interferences between reactions of various binder components make use of complimentary analytical techniques essential.¹¹

Fourier-transform infrared spectroscopy is another method that has been used for mortar analysis. As previously described in Practice Points Number 4, this method has the greatest utility for identifying inorganic materials, making it particularly suited to identification of organic additives.¹²

Chemical Analysis

Chemical analysis includes methods that range from simple acid digestion to relatively complex wet-chemical procedures. They have been used historically in the analysis of mortars to determine the contents of acid-insoluble material, soluble silica (indicative of an hydraulic component), calcium and magnesium oxides, sulfates, etc. Procedures such as loss on ignition are used to identify the presence of hydrated mortar species, as well as other minerals. With the exception of loss on ignition, chemical analysis procedures yield the percentage of a particular elemental oxide in an unknown. By-products of the analysis (for example, filtrates from acid-dissolution procedures) can be analyzed instrumentally using methods such as atomic-absorption spectroscopy and inductively coupled plasma to gather even more information about a mortar's chemistry. Ultimately, though, for wet-chemical methods to yield useful information about the starting com-

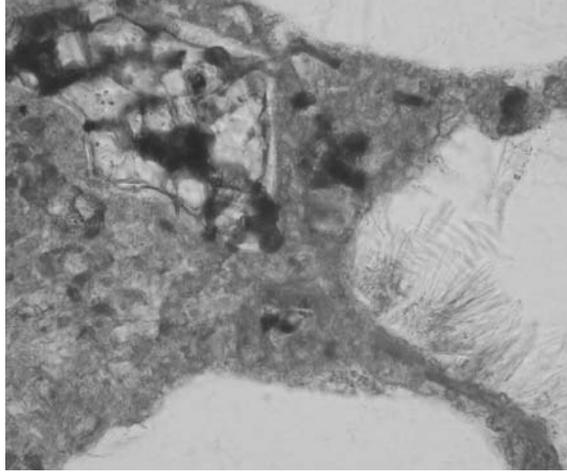


Fig. 4.

View of a portland-cement-based mortar in plane polarized light, 400x. The grain at the upper left of the photo is an unhydrated cement grain; the area to the right is a pore lined with ettringite. The white areas above and below are quartz grains. The binder consists of a mixture of calcium silicate hydrate, calcium hydroxide, and smaller grains of unhydrated cement.

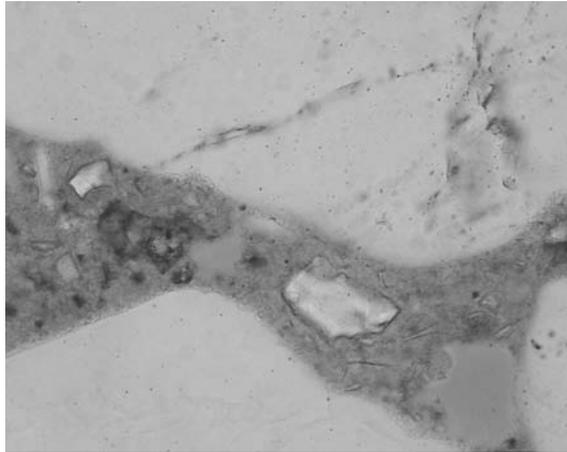


Fig. 5.

Stucco in thin section taken in plane polarized light, 100x. The grains at the top and bottom of the view are quartz; the small, angular white grain in the binder between the two quartz grains is a fragment of ground granulated blast-furnace slag. The dark particle to the left in the binder is unhydrated portland cement.

ponents of a mortar, some type of data analysis is required. This analysis might include either back calculation from standardized compositions of likely starting materials¹³ or knowledge of the starting materials gained through other analytical methods or documentation.

Acid Digestion

One chemical analysis method with which most preservation professionals are familiar is acid digestion. In fact, the use of simple digestion of crushed mortar in dilute acid as a suitable method for mortar analysis seems to be so embedded in the consciousness of preservation professionals that some additional discussion of its real utility is warranted.

As a chemical analysis method, acid-digestion analysis serves two useful functions. The first is determining the proportion by weight in the mortar of acid-insoluble material. Acid digestion of a crushed mortar sample is accordingly a component of most chemical-analysis protocols.

The second and more critical function of acid digestion of mortar is obtaining a sample of an original silica mortar sand for closer examination and matching to currently available resources. Yet even for this latter purpose, simple acid-digestion methods are flawed, as the process of grinding the mortar prior to digestion can crush the aggregate, altering both the shape and size of the grains. This problem is especially severe for hard mortars that are high in cement content. Such mortars are poorly dissolved by acid unless they are ground quite finely, which irreversibly alters the aggregate size and shape.

In and of itself, acid digestion provides no objective data on the composition of the binder, short of the mass of acid-soluble material it contains. The presence of carbonate-based acid-soluble aggregates (shells, coral sand, crushed limestone, or marble) make the method completely useless for any sort of compositional determination.

Advances in the field of mortar analysis have unequivocally demonstrated that the simple acid-digestion methods proposed by E. Blaine Cliver and H. Jędrzejewska are not suited to the general analysis of historic mortar.¹⁴ These methods are limited, in that they have no utility for mortars with acid-soluble aggregate, and Cliver's method is fundamentally flawed in the conclusions that can be drawn regarding original binder composition.

These two early methods relied on the fact that most mortars have acid-soluble binders and acid-insoluble aggregates. Cliver's proposed method was intended to be "simple, relatively accurate, and....done in the field at little expense and with only a basic knowledge of chemistry."¹⁵ He then goes on to describe the use of the method to differentiate construction periods for a building or to identify alterations. Jędrzejewska's method was intended to provide "a quick and easy method of technological classification of mortars" as the "usual methods of mortar analysis were...too detailed, lengthy, and expensive to be used for the routine examination of many hundreds of mortars."¹⁶ Interestingly, both authors reference other instrumental techniques that can be used to provide information about the composition of historic mortars. Cliver mentions X-ray diffraction and spectroanalysis; Jędrzejewska describes spectrographic methods, thermography, and the use of electron microscopy.

Cliver's and Jędrzejewska's methods were examined in a study by John Stewart and James Moore published in 1981, where they were used to determine the compositions of a suite of standard mortars of known composition.¹⁷ Unfortunately, Cliver's desire to provide an inexpensive field screening method was not successful. Cliver's method was refuted when it failed to correctly distinguish the compositions of any of the standard samples. However, the authors found that Jędrzejewska's method "meets its claim of being a simplified, semi-quantitative technique," which could "act as a rapid screening test to distinguish between those historic mortars which are non-hydraulic and those which may be hydraulic."¹⁸ Subsequent attempts to utilize Jędrzejewska's method suggest that the results are, in part, determined by the nature of the apparatus (Fig. 9). The procedure is temperamental in practice, yielding different results when performed using the same apparatus on the same sample by different analysts, and of limited utility in mortars that contain significant amounts of hydraulic material.

Analysis Protocols

Despite the complexity and widely varied nature of hardened historic mortars, there remains a desire on the part of many for a protocol for analysis of historic mortars. In its simplest form, a protocol is a standardized set of analytical procedures. Standardization of test methods and reporting results allows for comparison of analytical results between laboratories, thereby increasing knowledge and the understanding of materials.

Various analysis protocols have been proposed. The most familiar may be the ASTM C 1324. ASTM C 1324, "Standard Test Method for Examination and Analysis of Hardened Masonry Mortar," is a method that was first published by ASTM in 1996; the most recent revision was in 2005.¹⁹ The method consists of multiple analytical procedures that are supposed to be performed on a sample of mortar in sequential fashion. The primary analytical procedure is petrographic examination. Petrographic examination in this standard "refers to...light microscopy and to use of a petrographic microscope and a stereoscopic low power microscope."²⁰ XRD is included under petrographic examination and is "necessary for interpretations in calculating mortar composition."²¹

The petrographic examination is broken down into four sections: mortar, aggregate, paste, and air. The mortar is first examined intact, presumably microscopically under low power in a hand sample; XRD diffraction is indicated to be performed on the bulk sample (i.e., binder and aggregate). In mortars with acid-insoluble aggregate, the standard calls for breaking the sample to facilitate digestion of the paste (or binder, as it is more commonly called when referring to mortar) in dilute hydrochloric acid. The aggregate is washed, dried, and examined microscopically; it may be sieved, though the standard cautions that the sieve analysis may be skewed if the aggregate is broken when the sample is ground. The paste is examined using the methods of Practice C 856, "Standard Practice for Petrographic Examination of Hardened Concrete," which includes visual examination, stereoscopic (low-power microscopy) examination, and polarized-light microscopy.²² Air content is evaluated and estimated visually; the method of ASTM C 457, "Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete," is indicated as an option.²³

The results of the petrographic examination are used to determine which of the sub-procedures should be used. Sub-procedures consist of elemental analysis techniques for determination of silicon dioxide, calcium oxide, and magnesium oxide; determination of acid-insoluble content by acid digestion; determination of combined water and carbonate content by loss on ignition; and determination of magnesium hydroxide by DTA. The results of the sub-procedures considered in

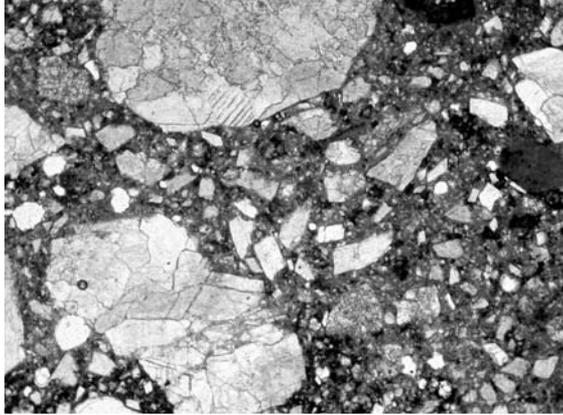


Fig. 6. Dolomitic mortar from the Charterhouse of Žiče in Slovenia in plane polarized transmitted light, 100x. Courtesy of Federico Carò, Research Fellow, Metropolitan Museum of Art, July 2007.

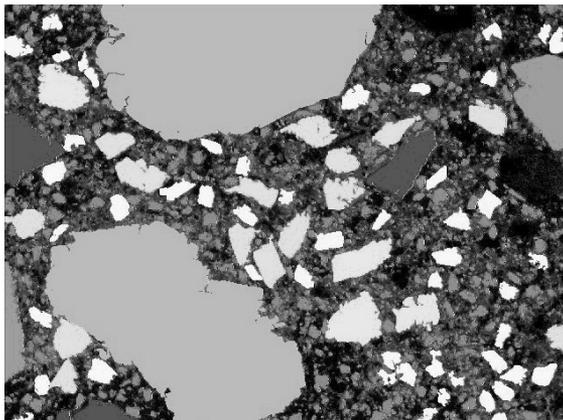


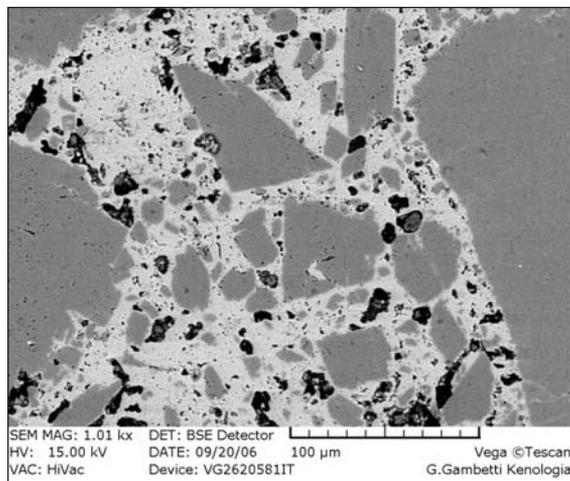
Fig. 7. A computer image generated from the photomicrograph in Figure 6 using digital image processing with image-analysis software. This software uses different colors to distinguish particle size. Courtesy of Federico Carò.

light of the petrographic analysis are used to determine the materials making up the mortar. The quantitative numerical data from the sub-procedures is then used to calculate the original proportions of the various materials. Unfortunately, these calculations are only possible for a limited suite of mortar types. The standard suggests that proportion calculations for other mortar types can be made "based on other detailed information from the petrographic analysis" but also indicates that "very old buildings probably contain mortars made using hydrated lime, or hydraulic lime or natural cement, which must be addressed on an individual basis."²⁴

More recently, a study funded by both a National Center for Preservation Technology and Training (NCPTT) grant and a Samuel H. Kress Foundation publication grant was developed by Elizabeth Goins. The objective of the work, initiated in 1998, was to develop a new protocol for the analysis of historic mortars. Two publications resulted. The first was a review of the literature, published in the RILEM-sponsored conference in Scotland.²⁵ The second was a draft-final report published through NCPTT.²⁶ The protocol consists first of visual analysis of the sample under low magnification. For samples where the aggregate is siliceous, the next step is acid digestion of a sample, followed by sieve analysis of the acid-insoluble material. Where the aggregate is determined to be soluble, modal analysis of a section is recommended. XRD is also suggested for aggregate determinations. The method for binder characterization is not specified, though XRD, modal analysis, and SEM/BSE are all mentioned. Determination of depth of carbonation by phenolphthalein is also recommended. Generally speaking, there seems to be a great deal of latitude in which methods are applied that makes this less of a protocol and more of a description of possible courses of action.

Fig. 8.

A backscattered-electron image of a polished thin section of mortar. Different shades of grey correspond to average atomic number, allowing distinction between areas of different composition in a sample. Courtesy of Federico Carò.



Perhaps the most comprehensive protocol for compositional analysis was presented by the RILEM technical committee TC 167-COM in its final report.²⁷ The proposed protocol, which is outlined in two flow charts, utilizes virtually every instrumental analysis method previously discussed (as well as several that were not).²⁸ If followed in its entirety, the protocol would return a tremendous amount of data on mortar composition.

Not surprisingly, most of these protocols rely on petrographic analysis as the jumping-off point for the analysis, supplemented by XRD analysis and chemical analysis. However, the significant drawback to all these protocols is the absence of any data illustrating their effectiveness when tested against a suite of samples of known composition. They also lack any sort of guidance as to how the analytical data are to be interpreted for historic mortars of unknown composition. A comparison of these protocols similar to that performed by Stewart and Moore for the Cliver and Jedrzejewska methods would provide great insight into which of these protocols might actually have utility.

Conclusions

The sum total of research into the analysis of the composition of historic mortars to date clearly indicates that positive identification of the original components of a mortar, particularly the binder, is a complex task requiring quantitative analytical methods executed by experienced analysts with a good understanding of both the reaction chemistries involved and historic materials. Even under the best circumstances, positively identifying the original components and particularly the proportions of the original binder materials may not be possible.

Another critical observation is that there is no agreement as to the “right” approach for mortar analysis. Aside from a general agreement as to the utility of petrographic analysis, and in particular PLM, in understanding historic mortars, researchers continue to utilize a range of techniques to broaden their understanding of historic mortar compositions. Nevertheless, many practitioners still strongly desire a single, standardized analysis protocol that provides results that can be compared to one another — even though this is unlikely to be achieved in practice. This desire was likely the impetus in 1996 for the development of ASTM C 1324 and the rationale for Goins’s work.

Yet the fundamental reality in the field of preservation is that there is no single reason for performing mortar analysis that makes such a protocol necessary. Frankly, despite the dictate of the Secretary of Interior’s Standards for Rehabilitation, knowing the precise original composition of the mortar may not be necessary. More than 20 years ago, both Jedrzejewska and Morgan Philips directly confronted the fact that a significant reason for doing mortar analysis is to understand what kind of replacement mortar will be appropriate. The international community has acknowledged this fact as well by referring to their work as characterizing mortars with respect to their repair. Methods and protocols used for mortar analysis must be adaptable to the project needs and must provide the maximum information for the minimum cost, as cost is typically the overriding factor in determining what kind of analysis is done or if there is even analysis at all. The issue of why and how to obtain mortar analysis services will be addressed in the next Practice Point in this series.

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Notes

1. Lorraine Schnabel, “Mortar Analysis Part 1: Mortar-Making Materials,” Practice Points Number 5, *APT Bulletin* 39, no. 1 (2008): 1–4.
2. Elizabeth S. Goins, *Standard Practice for Determining the Components of Historic Cementitious Materials*, Publication No. 2002-20 (Washington, D.C.: U.S. Dept. of the Interior, National Park Service, National Center for Preservation Technology and Training, 2004): 45–68. This document is available for free download from the NCPTT Web site at www.ncppt.nps.gov/pdf/2002-20.pdf.
3. Kara Dotter, “Characterization of Historic Lime Mortars” (master’s thesis, Graduate School of Geological Sciences, University of Texas, Austin, 2006).
4. C. Groot, G. Asheall, J. Hugues and P. Bartos, “Characterisation of Old Mortars with Respect to Their Repair: A State of the Art,” in *State-of-the-Art Report of RILEM Technical Committee 167-COM: Characterisation of Old Mortars with Respect to Their Repair*, RILEM Report No. 28, 2007 edition, ed. C. Groot, G. Asheall, and J. Hughes (France: RILEM Publications S.A.R.L., 2004–2007), 1–6. All parts of this document are available to view online at www.rilem.net/reports.php. Click on the report title to view the document online.
5. Donald St. John et al., *Concrete Petrography, A Handbook of Investigative Techniques* (London: Arnold, 1998), chap. 3. Though the handbook was specifically developed for use in examination of concrete specimens, many of the sample-preparation techniques and methods have utility in the examination of hardened mortars.
6. J. Elsen, “Microscopy of Historic Mortars—A Review,” *Cement and Concrete Research* 36 (2007): 1416–1424.

7. Information on the principles and procedures of point counting can be found in petrology texts, particularly sedimentary petrology texts. One source is John S. Galehouse, "Point Counting," in *Procedures in Sedimentary Petrology*, ed. Robert E. Carver (New York: Wiley-Interscience, 1971), 385–425.

8. F. Carò, A. Di Giulio, and R. Marmo, "Textural Analysis of Ancient Plasters and Mortars: Reliability of Image Analysis Approaches," in *Geomaterials in Cultural Heritage*, ed. M. Maggetti and B. Messiga (London: Geological Society, Special Publications, 2006): 257, 337–345, and F. Carò and A. Di Giulio, "Reliability of Textural Analysis of Ancient Plasters and Mortars through Automated Image Analysis," *Materials Characterization* 53 (2004): 243–257. Past personal experience suggests that image-analysis techniques can underestimate the aggregate proportion where a mortar contains abundant fine-grained material.

9. Normally thin sections are covered with a thin square of glass called a cover slip to protect the sample.

10. Joshua Freedland et al., "Through a Lens Clearly: Instrumental Analysis as Applied to Architectural Materials," Practice Points Number 4, *APT Bulletin* 38, no. 4 (2007): 1–4.

11. B. Middendorf et al., "Mineralogical Characterisation of Historic Mortars," in *State-of-the-Art Report of RILEM Technical Committee 167-COM*, 30–32.

12. Freedland, 3–4.

13. For example, the oxide composition of modern and even older portland cements is not only well understood but also tightly controlled.

14. E. Blaine Cliver, "Tests for the Analysis of Mortar Samples," *APT Bulletin* 6, no. 1 (1974): 68–73. H. Jedrzejewska, "Old Mortars in Poland: A New Method of Investigation," *Studies in Conservation* 5, no. 4 (1960): 132–138.

15. Cliver, 68.

16. Jedrzejewska, 132.

17. John Stewart and James Moore, "Chemical Techniques of Historic Mortar Analysis," in *Mortars, Cements and Grouts Used in the Conservation of Historic Buildings* (Rome: ICCROM, 1981), 297–310.

18. Stewart and Moore, 302.

19. ASTM International "Standard Test Method for Examination and Analysis of Hardened Masonry Mortar," ASTM C 1324-05, in *Annual Book of ASTM Standards*, vol. 4.05, *Chemical-Resistant Nonmetallic Materials; Vitrified Clay Pipe; Concrete Pipe; Fiber-Reinforced Cement Products; Mortars and Grouts; Masonry; Precast Concrete* (West Conshohocken, Pa.: ASTM International, 2008).

20. ASTM International, ASTM C 1324-05, 2.

21. Ibid.

22. ASTM International, "Standard Practice for Petrographic Examination of Hardened Concrete," ASTM C 856-02, in *Annual Book of ASTM Standards*, vol. 4.02, *Concrete and Aggregates* (West Conshohocken, Pa.: ASTM International, 2003).



Fig. 9. Gas-collection apparatus for acid-digestion analysis based on the original design by H. Jedrzejewska as described in his 1960 article. Courtesy of Lori Aument, John Milner Associates, Inc.

23. ASTM International, "Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete," ASTM C 457, in *Annual Book of ASTM Standards*, vol. 4.02, *Concrete and Aggregates* (West Conshohocken, Pa.: ASTM International, 2003).

24. ASTM International, ASTM C 1324-05, 6, para 10.2.

25. Elizabeth S. Goins, "A New Protocol for the Analysis of Historic Cementitious Materials: Interim Report" in *Historic Mortars: Characteristics and Tests, Proceedings of the International RILEM workshop, PRO 12*, ed. P. Bartos, C. Groot, and J. J. Hughes (France: RILEM S.A.R.L., 2000): 71–80.

26. Goins, "Standard Practice," 30–33.

27. B. Middendorf, J. J. Huges, K. Callebaut, G. Baronio, and I. Papyanni, "Mineralogical Characterization of Historic Mortars" and "Chemical Characterization of Historic Mortars," in *State-of-the-Art Report of RILEM Technical Committee 167-COM*, 21–54.

28. Middendorf, 36 and 53.

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