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Petrography: Distinguishing Natural Cement from Other Binders in Historical Masonry Construction Using Forensic Microscopy Techniques

ABSTRACT: Petrography is a microscopy method providing a revealing view of inorganic materials. Hydraulic cements rarely react completely and telltale residuals are detected by the petrographer. Combined with accurate chemical analysis, the petrographic examination not only identifies the original components but often allows for a quantitative assessment of material proportions. Such data are crucial for the proper conservation and rehabilitation of historic masonry structures. Portland cement, lime, and natural cement in any combination may be present in historic American masonry construction. A comprehensive petrographic examination serves as the basis for the successful creation and implementation of an historically accurate project specification. This paper provides insight into how various binders are properly distinguished. While technical data will be offered, the goal is to provide the architect or conservator with a general understanding of the methods employed by the petrographer and to demystify the often complicated mortar analysis report.

KEYWORDS: mortar analysis, natural cement, petrography, Rosendale, historic binders, masonry conservation

Introduction

Materials analysis is often perceived by the construction industry as the proverbial black box; a randomly chosen sample is placed in one end and a perfectly unequivocal result appears at the other. It is the workings inside this mythical contraption that are usually misunderstood. The scientific language accompanying materials examination reports may give the false impression that the sample has undergone an invariable procedure that would produce identical results no matter which black box was chosen. Fortunately, the preservation industry has a sympathetic appreciation for the forensic materials scientist's task. All preservation projects have a forensic component and all involved are familiar with the detective work that is usually required. As with criminal investigations, a great part of the investigation's success relies on the intuition, experience, and approach of the detective. Nevertheless, the particular skills and experience the materials analyst possesses are rarely understood by others in the preservation industry and blind faith is too often the governing criterion for choosing a laboratory.

As the volume in which this paper appears is a special issue dedicated to the American natural cement industry, a portion must focus on identifying that particular binder in historical construction. The binder in cementitious construction materials is the most dynamic of the system, expected to chemically react and convert to a new material and as such the most difficult to identify in older construction. Most conservators and preservation professionals would be surprised to learn of the paucity of modern technical literature regarding the properties and identification of natural cement. The primary goal of this paper is to share with other analysts the criteria and approaches this investigator has learned to utilize in examinations of historical masonry construction. This summary contribution aims to add something to the practitioner's toolbox and encourage further research into this resurgent industry. However, an additional goal is to give some insight to those not privy to the methods of the materials scientist and hopefully allow for more effective and educated partnerships between the preservationist and the materials analyst.

TABLE 1—*Typical chemical analyses of two mortars.*

Chemical Data	Sample A[a]	Sample B[b]
SiO_2	3.05	3.06
CaO	11.86	10.64
MgO	2.03	2.09
Al_2O_3	0.77	1.76
Fe_2O_3	0.58	0.82
Insoluble residue	74.27	69.65
Loss on ignition	6.68	11.26
Total	99.24	99.28
Proportions	1:1:6	1:0.7:5.6
Type (ASTM C 270)	N	N

[a]Sample A is a fictitious Type N portland cement-lime mortar.

[b]Sample B is an actual analysis from a natural cement mortar sampled from a stone masonry pier at High Bridge over the Harlem River in New York City constructed circa 1848.

Chemical Analysis

In the United States, mortar analysis methods are provided for in ASTM C 1324 [1]. This relatively new document calls for both the analytical skills of the chemist and the observational techniques of the microscopist or petrographer. What seems to go unnoticed is how this collaboration must result in a set of observationally based assumptions used to interpret the hard numbers of the chemical analysis. The required forensic skills are explicit in the document and the standard reads as a set of guidelines and suggestions for interpretation rather than an algorithmic methodology. While a great deal of interpretative freedom is left to the analyst, the rules of the chemical analysis may seem to represent the comfortable and infallible black box. When blind adherence to the chemical rules are applied by a laboratory and these are provided to a restoration contractor insensitive to historical materials, predictable but potentially disastrous results are likely.

The chemical procedures require an acid digestion to separate the sand from the binder matrix. Ideally, the sand is recovered as a wholly insoluble component while the binder matrix goes into solution. The solution containing the dissolved binder is then analyzed to determine elemental concentrations. Water and carbon dioxide are measured separately by igniting the sample in a furnace and determining weight losses at specific temperatures. In most cases, these procedures isolate and partition the components reasonably well. A presentation of typical chemical analyses is given in Table 1. What is important to note is that the results comprise elemental percentages rather than mortar component percentages. In order to come up with raw material ratios a number of assumptions must be made. The interpreted mortar design is only as good as the assumptions made about the original components even if the chemical analysis is highly accurate.

When considering binder components, the first assumption involves the partitioning of the measured elements into the original raw materials. Consider the chemical analysis of Sample A shown in Table 1. Let us assume the mortar is identified as a contemporary cement-lime mortar. While an oversimplification, the important measurements to consider here are silica (SiO_2) and calcium oxide (CaO). Modern portland cement contains both silica and calcium oxide at approximately 21 and 63 %, respectively. Hydrated lime contributes only calcium to the analysis. If these assumptions are valid, one can divide the measured silica weight by 21 % and determine the original cement weight. This would result in 14.5 % cement by weight for Sample A. Given this portland cement proportion, we would need to deduct 9.2 % of the measured calcium oxide to partition into the cement. The remaining calcium oxide must then belong to the hydrated lime. Similar calculations may be made for magnesium oxide which is present in dolomitic limes. With some simple manipulation based on molecular weights, these oxides are then mathematically converted resulting in 6.2 % hydrated lime by weight. Of course, masons do not mix mortar by weight so we must make some more assumptions to convert these weights to volumes. If we assume that cement and lime have densities of 94 and 40 lb per cubic foot, respectively, it is a simple matter to divide the weight percentages by density resulting in a one to one volume proportion. Similar manipulations for the aggregate result in a classic Type N cement-lime mortar at a 1:1:6 cement to lime to sand proportion. It should be clear that there is some error built into these conversions. For instance, if the original cement contained 2 % more silica as might be typical in a white portland cement, there could be a 10 % error in the calculated cement weight.

FIG. 1—*High Bridge over the Harlem River in New York City. The photograph was taken before 1927 when masonry piers over the river were removed to accommodate a steel span to allow for larger naval ships to pass. The picturesque New York landmark was constructed using Rosendale cement manufactured several tens of miles north along the Hudson River.*

Though the chemistry assumptions alone may produce a significant error, these pale in comparison to those produced by misidentifying the original cementitious components. The analysis given for Sample A is admittedly fictitious and represents an idealized Type N portland cement-lime mortar completely manufactured by the author. The data presented for Sample B are an actual chemical analysis using the same techniques described above. Interestingly, the data are very similar to that of Sample A with some very minor differences. In fact, if we were to go through the same mathematical procedures we would find the mortar to be a $1:0.7:5.6$ proportion by volume also characterized as a Type N cement-lime mortar. In actuality, the mortar was sampled from a stone masonry pier at High Bridge over the Harlem River in New York City constructed circa 1848 as part of the city's aqueduct system (Fig. 1). Original records show that the bridge was constructed using Rosendale natural cement produced in upstate New York [2] and petrographic examinations performed by the author are consistent with historical data. Clearly, there is nothing in the chemical analysis that would uniquely characterize the material as a natural cement mortar. Other methods are required to place the chemical data into a proper context and allow for an accurate design estimate.

Mineralogical Methodology

When requesting a mortar analysis on an historic property, the conservator should ensure that some method will be used that identifies the actual binder components in addition to producing chemical data. This is particularly important for structures built in the mid- to late 19th and early 20th centuries where any combination of binders may be present. Regardless of the method chosen, it must at the very least be capable of identifying mineral species and ideally should also be able to discern microstructural evidence that aids in distinguishing the various binders. X-ray diffraction is an excellent method for determining mineral phases but it cannot image the binder residuals that are almost always present within the mortar matrix. Scanning electron microscopy is a perfect tool for imaging microstructure and when properly calibrated may also quantify mineral compositions. However, the analysis may be prohibitively expensive for routine examinations. Both tools provide excellent supplementary data where other methods fail but the workhorse for mineral identification is the polarized light microscope. Petrographic methods using polarized light microscopy are over a century old and are adopted from the geological sciences [3]. Advantages of the technique include its ability to image binder residuals and determine mineralogy, and its relatively low expense as a laboratory tool.

Polarized light microscopy relies on the principle that plane polarized light passing through a crystalline material will usually split into two perpendicular rays traveling at different velocities. The velocity difference is a function of the crystallography and orientation of the mineral. As the light rays reach a second polarizing film, they are resolved back into the same plane. Due to the difference in the wave

speeds these two rays will interfere with one another producing light of a particular color. The color differences are used by the petrographer to glean quantitative information about the mineral species being viewed. Petrography is a methodology defined by many scales of analysis one of the most important being that of the polarized light microscope and its etymology is "rock pictures." The "picture" part of the root represents an important feature of the microscope. With adequate sample preparation, the microstructural relationships of the binders and aggregates are readily viewed by the petrographer to a resolution measured in the thousandths of a millimetre. Combined with the quantitative crystal data, a tremendous amount of information is available to the analyst in an intuitive pictorial manner that does not present itself in many other analytical techniques.

There are several ways of preparing a mortar specimen for microscopic examination. The most commonly employed in petrographic laboratories is the powder mount. The procedure costs pennies and preparation time is measured in tens of seconds. The sample is crushed and sieved and a fraction of a milligram is placed on a glass slide in an oil immersion. When looking for residual binders, the petrographer must hunt these mortar crumbs for any evidence of unhydrated or uncarbonated binder minerals. The method is perfectly acceptable for routine analyses of contemporary mortars where all that needs to be determined is whether the portland cement is white or gray and whether or not crushed limestone is present as the lime addition. However, residual lime grains and natural cement residues in historic materials are exceptionally difficult to detect using this method.

Thin sectioning is the preferred method for examining historical materials microscopically. The material is slabbed and wafered and impregnated in a low viscosity epoxy. The mounting surface which may represent as much as 3 in^2 is sequentially ground, rough polished, and epoxy-mounted to a glass slide. The mounted wafer is then trimmed and milled until the surface to be examined is one-thousandth of an inch in thickness and essentially transparent. The material may now be examined in an unadulterated state with every microscopic particle in its original position. Components that represent a trace fraction may be easily detected with their boundaries intact and abundances readily estimated. The disadvantage of the method is the increased cost and turnaround time. Considering the schedule and budget of conservancy projects where replacement-in-kind is a high priority; these disadvantages hardly seem significant when weighed against the quality of the obtained results.

Detecting Binders Petrographically

The key to binder identification lies in the unreacted residuals. One would think that older mortars should not contain unreacted binders. In fact, many older materials exhibit more residual cement or lime than even contemporary materials of several years age. The coarser grinds of older binders combined with the generally lower quality control in production results in grains large enough to survive decades and even centuries in most service environments. It is not uncommon to find unreacted grains visible to the naked eye (Fig. 2). These coarser grains, even when microscopic, provide abundant evidence for the petrographer to sort through. The various cementitious materials have sufficient differences in their raw feeds, burning temperatures, mineral phases, and microstructure that a well trained petrographer should have a reasonably good chance of distinguishing them in thin section.

Portland Cement

Portland cement is a distinctly different product than either lime or natural cement and this difference is easily detected petrographically. Portland cement is manufactured from ground limestone and shale and is formed through a process known as clinkering. This means the raw feed is brought to temperatures in excess of 1400°C, sufficient to cause a virtually complete reaction to hydraulic mineral species. The phases that are important in the investigation of historic materials include two types of calcium silicate known as alite and belite and an iron-bearing species known as ferrite. Portland cement manufactured in the late 19th and early 20th centuries was typically less finely ground than in the later 20th century with individual particles often approaching a large fraction of a millimetre in size. So while all the constituents are reactive in the presence of water, it is very common to find an abundance of relatively large, partially unreacted cement grains. In older cements, the belite phase is most obviously abundant forming clusters of small subrounded crystals often described by petrographers as the "bunch of grapes" texture (Fig. 3). The crystals themselves are clearly defined and the crystallographic properties under polarized light are well
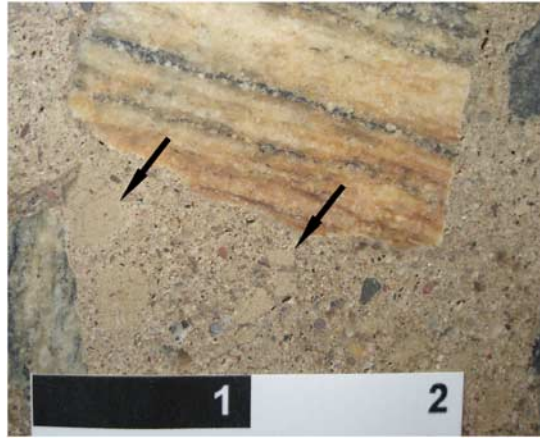
FIG. 2—*Close-up photograph of a polished cross section of concrete produced with Rosendale natural cement. The arrows indicate visible tan colored lumps of residual cement. Large unreacted or partially grains such as these are relatively common in historic construction materials and greatly aid the forensic petrographer in identifying the binder. The included scale is in inches.*

known to concrete petrographers [4]. Small clusters of belite may be detected in trace quantities in hydraulic limes but the difference in abundance should preclude any misidentification. With the exception of white portland cements, the belite phases are surrounded by a brown-colored crystallographically indistinct ferrite phase. Ferrite is not a mineral with a strict proportion of elemental constituents and is a phase that will likely form if any iron is present in burned materials containing calcium and aluminum. As such, it is almost sure to be present in a cement of any hydraulicity and should not be used to distinguish between the various binders. What clearly distinguishes portland cement from other historical binders is the presence of alite (Fig. 4). Alite is not a thermodynamically stable phase at temperatures below approximately 1300°C [5]. Nevertheless, the phase may be difficult to detect in older portland cements due both to its lower original abundance and its higher reactivity than belite. The particular structure of the well defined belite clusters should be sufficient to identify the cement but the identification of alite ensures the petrographer that a clinkered cement is the binder present.

If a fresh sample can be extracted sufficiently away from environmental exposure, additional information may be gleaned from the hydration products rather than the unhydrated residual particles. Portland
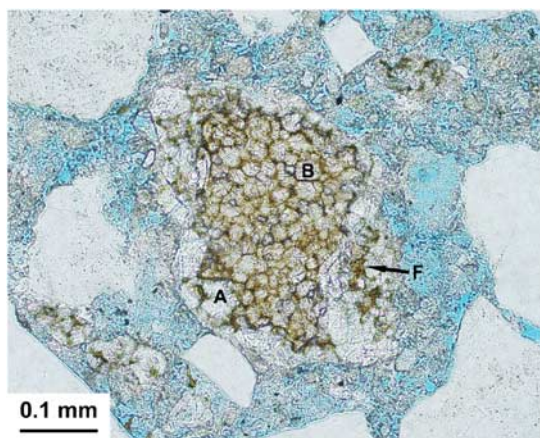

FIG. 3—*Plane polarized light photomicrograph of a residual portland cement particle typical of those detected in early 20th century materials. Petrographers refer to these relicts as having the "bunch of grapes" texture. The texture is produced by agglomerates of the calcium silicate mineral belite (B). Large groupings of small belite crystals are typical of older portland cements. Alite (A) is another calcium silicate and occurs only when clinkering temperatures are obtained. Its presence positively identifies a clinkered cement as opposed to natural cement or lime. The brown-colored ferrite phase (F) is common in portland cement but may be present in any binder containing calcium, aluminum, and iron.*
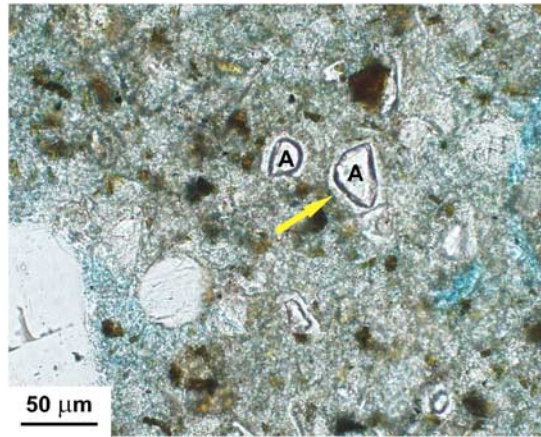
FIG. 4—*The mineral alite (A) is found exclusively in clinkered cement products. Note how the relict grains appear to stand out above the surrounding matrix. This is a function of the slower light velocity through the mineral as opposed to surrounding materials. This "relief" helps identify alite. The arrow indicates a colorless halo around the grain that represents a hydration rim produced during the reaction of alite with added mix water.*

cement reacts to form a combination of hydrates including calcium hydroxide. While the hydroxide is the primary constituent of unreacted lime, its form is distinctly different when a product of portland cement hydration. Crystals are orders of magnitude larger than in hydrated lime. The size difference is easily distinguished under the petrographic microscope in contrast to instrumental methods such as X-ray diffractometry where the mineral is detected but not imaged.

Limes

Lime mortars of any type are quite different in both composition and structure. In fact, there is little danger of mistaking a lime for a portland cement when appropriate petrographic techniques are applied. Whether a hot-mixed lump lime, fat lime putty, or eminently hydraulic lime, the principal component of the raw material is calcium hydroxide. Manufacturing processes are quite different than for portland cement. A limestone of variable purity is "calcined" or brought to a temperature just high enough to drive off the carbon dioxide present in the original limestone. This temperature is much lower than the clinkering point and is typically less than $1000°C$. The primary product is calcium oxide or free lime. However, this is a highly unstable compound and an additional step called slaking is required. There are a variety of slaking methods but simply put, it involves the addition of water to convert the oxide to calcium hydroxide. With some exceptions, the lime curing process does not involve hydration but rather carbonation. Over longer time periods than required for cement hydration, the lime begins to absorb carbon dioxide from the atmosphere, finally converting the binder back to calcium carbonate. The process is essentially one that produces an artificial limestone returning the product back to its approximate original form.

As with portland cement, residual lime grains are usually apparent even in lime mortars many centuries old. These rounded white lumps are distinctive and a simple powder scraping placed under the microscope may be sufficient for a tentative identification if the grain is macroscopically visible. But thin section examination is required to positively identify residual grains smaller than one millimetre. The difficulty in using simple powder mount techniques for lime identification is that the relict grains and the carbonated matrix are all composed of very fine-grained calcium carbonate as are the carbonated portions of hydraulic cement pastes. The presence of lime is revealed in the microstructural relationship between the residual grains and the surrounding matrix. Even though compositionally identical, the relict grains often have a fairly distinct boundary and a slightly denser character than the surrounding matrix (Fig. 5). Differential shrinkage between the two components often causes differing microscopic crack structures betraying the location of the relict grain. Additionally, the higher porosity and characteristic microscopic shrinkage cracking of lime mortars are blatantly obvious in thin section when no cementitious binders are present. Once these are detected, the chemical analysis may be used to estimate the original mix design where lime is present with another binder.
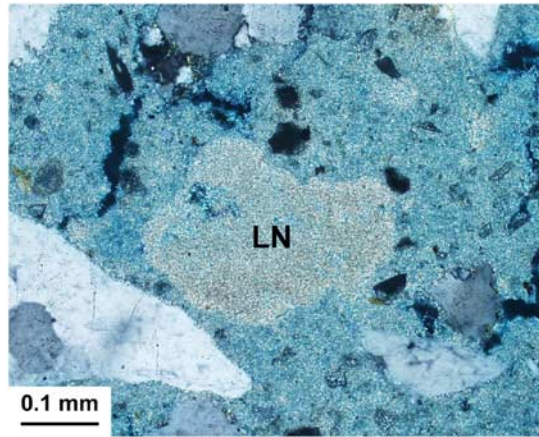
FIG. 5—*A residual lime nodule (LN) is depicted in this cross polarized light photomicrograph. The cream coloration of the grain identifies the carbonate that is a function of the curing process. The surrounding matrix is also carbonated but appears more blue-colored due to the blue-dyed epoxy impregnation that highlights the more porous areas. In powder mount, this grain would not be distinguished from any other carbonated cementitious material. Thin section preparation allows the petrographer to detect the subtle boundary between the lime grain and the otherwise carbonated matrix.*

Further complicating the lime story are the more hydraulic varieties. These begin as "dirtier" limestones containing clay, quartz, and other minerals in smaller proportion. At the calcining temperature, the aluminum and silicon present in these other mineral phases will begin to combine with the free lime to form hydraulic calcium aluminates and calcium silicates. These minerals may be identical to those present in true hydraulic cements. In many cases, the original silicate minerals may not fully react and it is not unusual for the petrographer to observe what appears to be normal geological quartz. However, these grains usually contain expansive crack structures and a rim of either higher temperature varieties of quartz or hydraulic calcium silicate. Nevertheless, their appearance within the boundary of a petrographically identified lime grain precludes the possibility that they derive from either a cement component or the aggregate (Fig. 6).

Natural Cement

All of the binder products discussed so far are familiar to most petrographers. On the other hand, natural cements have received much less attention in previous decades and many examples go unrecognized or
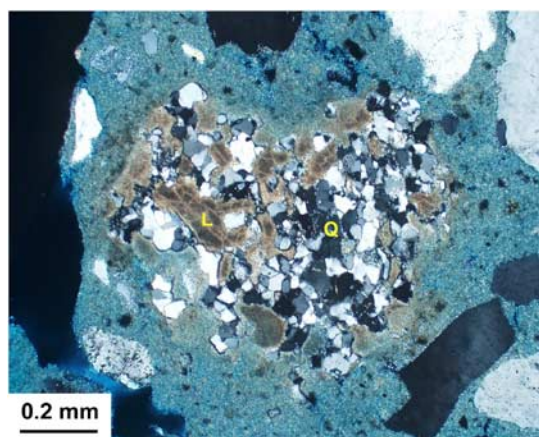

FIG. 6—*This cross polarized light photomicrograph illustrates a lime nodule representing a more hydraulic type of lime. The original limestone portion of the rock has been calcined and converted to the tan-colored lime (L) that has since carbonated. However, gray-colored grains of quartz (Q) are nearly unaffected by the burning process and retain their geologic character. The higher silica (or quartz) content of the lime identifies it as having some potential degree of hydraulicity.*
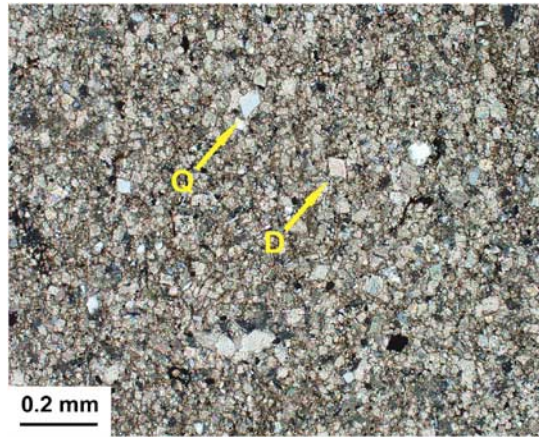
FIG. 7—*This cross polarized light photomicrograph illustrates the mineralogy and texture of the lower member of the Rondout Formation used in the production of Rosendale cement. Rhombic-shaped grains of dolomite (D) constitute a large portion of the rock. Gray-colored grains of quartz silt (Q) are evenly dispersed throughout the carbonated matrix. The overall darker brown coloration to the matrix is produced by clay minerals. The silica present in the quartz and the alumina contributed by the clays are responsible for the hydraulic properties of natural cement and distinguish it from other calcined limes.*

misidentified as a result. The situation is complicated by the fact that natural cements may be highly variable in microstructure. Fortunately, there are several distinctive microtextures that allow for a positive identification and petrographic techniques remain one of the best tools for distinguishing these features. Before describing these textures, it helps to have an understanding of the raw materials and burning processes involved in natural cement manufacture.

While admittedly an oversimplification, natural cement may be thought of as residing within the continuum between portland cement and hydraulic lime. The composition of the raw materials are more similar to those of portland cement containing higher proportions of silica and aluminum. These are the elements that when combined with calcium will form the hydraulic minerals. However, the burning temperatures for natural cements were closer to the calcining temperatures of limes and were never clinkered unless accidentally overburned. The Rosendale district cements manufactured in New York State between the 1820s and 1970s deserve special attention, as these represent a majority of American-made natural cements. These were produced from carbonate rocks of the Rondout Formation along the Hudson Valley. The raw materials used in their manufacture include two layers of dolostone rock between which a thin unproductive limestone is found [6]. Dolostone is composed of a magnesian carbonate as opposed to the calcium carbonate found in raw materials for fat lime. The combination of relatively low burning temperatures with the distinctive mineralogy allows for a distinctive set of microstructural features unique to natural cements such as the Rosendale. Historical literature suggests that a large majority of American natural cements were mined from dolomitic sources thus distinguishing them from all portland cements as well as European Roman cements [7]. Rosendale cements are therefore considered representative of the majority of 19th century American hydraulic masonry cements. Preliminary research into other materials such as those produced in Shepherdstown, WV appears to support this generalization.

Figure 7 illustrates the typical microstructure of the lower member of the Rondout Formation in its raw state. The bulk of the rock comprises carbonate material including rhombic-shaped dolomite crystals. In addition, dispersed grains of quartz and finely divided clays are relatively evenly distributed throughout the rock. Were this fine-grained rock to have been clinkered, much of the original texture would have been destroyed and replaced with relatively coarse-grained hydraulic species similar to those observed in portland cements and calcium aluminate cements. Instead, the rock was burned at temperatures closer to calcining temperatures. While this was sufficient to form hydraulic species, the obtaining time and temperature were not sufficient to completely rearrange the original microtexture of the rock. Reactions occurred across grain contacts without complete decomposition of the original constituent minerals.

Even before the individual binder grains are detected, the distinctive matrix produced by natural cement curing may be apparent. Portland cement pastes are homogeneously isotropic or dark-colored where cementitious gels have formed, broken only by thin dispersed grains of calcium hydroxide which
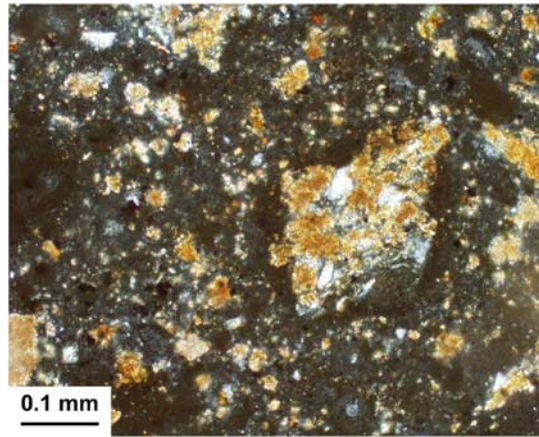
FIG. 8—*Natural cements may be thought of as containing both hydraulic as well as lime-type species. The mineralogy and texture shown in this cross polarized light photomicrograph is typical of natural cements. Portions of the rock were calcined during the burning process and essentially behave as lime. Carbonated material appears as bright-colored areas and represents calcined dolomite that has carbonated during the curing process. Dark areas of isotropic material represent hydraulic areas that have undergone hydration. This petrographic character is diagnostic of natural cement where fresh areas that have not been exposed to the environment are examined. This sample derives from a partially hydrated cement barrel found in the Widow Jane Mine in Rosendale, NY [8] believed to represent material produced during the early 20th century.*

appear bright-colored. Lime matrices are homogeneously carbonated appearing very bright in polarized light. Microscopic shrinkage cracking as well as higher porosity are also hallmarks. Natural cement matrices are distinctively different. Both hydraulic as well as lime-type species are present in raw natural cement and these are heterogeneously distributed at the micron scale of observation. Under polarized light, the matrix of natural cements often appears spotted with dark isotropic areas broken by bright but dense carbonated regions. While these textures may be highly varied, there will usually be areas in thin section that have overall features that are not easily attributed to either portland cement pastes or lime matrices (Fig. 8).

The residual binder grains are where the most unambiguous evidence exists. Our laboratory obtained samples of both the lower and upper members of the Rondout Formation from Rosendale, NY and fired the samples in a furnace for 24 h at a variety of temperatures. Figure 9 illustrates one of the distinguishing features when viewed petrographically. At temperatures as low as 600°C, iron contained within the individual dolomite crystals begins to migrate out, lining the crystals with an iron-bearing phase. By the time calcining temperatures are reached, the dolomite has completely decomposed leaving behind calcium oxide and magnesium oxide. Even though the original mineralogy can no longer be identified, the original rock texture is largely preserved. This is one of the diagnostic features of natural cements observed in actual historical construction. In many cases, after a century or so of exposure, the calcined dolomite may carbonate again. However, the iron-rich outline is preserved betraying the texture of the original raw feed (Fig. 10). The cement residuals may appear to the petrographer as an odd carbonate rock but the lack of crystallinity within the dolomite structure in combination with the iron-rich lining should signal the analyst that the grains are not part of the aggregate fraction.

The dispersed quartz grains and other sand-sized silicate minerals also aid in distinguishing natural cements. These tend to retain most of their original texture when burned at calcining temperatures (Fig. 11). As temperature is increased, quartz begins to transition through a number of different structural forms. At temperatures below the calcining temperature a slight adjustment of the silicate bonds produce a more open structure which results in a bulk volume expansion. This may cause the quartz to develop internal cracks that are microscopically visible. These are best observed in cements produced from the upper member of the Rondout Formation which contains coarser grained quartz than the lower member. At temperatures approaching the calcining temperature, quartz will convert over to a different crystallographic form known as tridymite. At this point, the petrographic properties are significantly different than those of the original quartz grains. Tridymite rims around original quartz grains may be detected by their
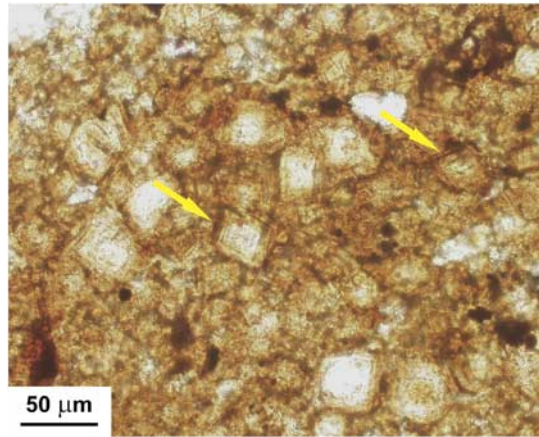
FIG. 9—*Our laboratory obtained samples of the Rondout Formation from Rosendale, NY and burned them at various temperatures for 24 h in a laboratory furnace. This plane polarized light photomicrograph illustrates lower member rocks burned at 600°C. The original rhombic-shaped dolomite crystals are still detected in their original positions and the rock fabric is not altered. However, iron contained within the dolomite crystals has migrated out to form a new iron-bearing species. The arrows indicate the reddish colored iron-bearing minerals that outline the original carbonate minerals.*

crystallographic properties in both natural cements as well as limes especially when underburned. Finally, as the surrounding carbonate minerals calcine and convert to oxide forms their composite elements become available for combination with the silicate and hydraulic calcium silicates may form. Belite is the most likely phase to form and this is the same mineral comprising the bulk of historical portland cements. However, the microtexture is so significantly different that when viewed using polarized light microscopy, there should be no mistaking these silicates for clinkered forms. In clinkered cements, the belite phase will be well developed and display the "bunch of grapes" morphology (Fig. 3). Belites in natural cements will be much less well developed and surround a core of petrographically identifiable quartz.

These partially reacted grains may pose a problem for petrographers when the rims have reacted as the grains have a nearly identical morphology to that of alite. This is especially problematic in cements manufactured from the lower member of the Rondout Formation in Rosendale as well as some Round Top natural cements produced in Maryland where quartz silt is present in the size range of typical portland cement minerals (Fig. 12). This is where careful use of the polarized light microscope comes into play. Due to the very different velocities of light through quartz and alite, the optical properties should betray
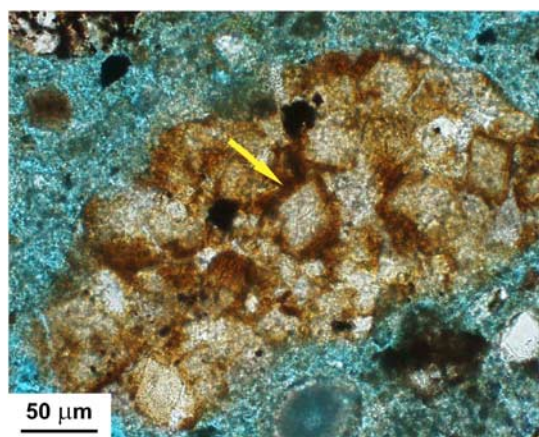

FIG. 10—*This plane polarized photomicrograph illustrates a residual grain of Rosendale natural cement circa 1850. Rhombic-shaped crystals of calcined dolomite are surrounded by reddish rims of an iron-bearing mineral phase (arrow). Note the similarity to the laboratory produced cement depicted in Fig. 9. This microtexture is diagnostic of natural cements produced from dolomitic raw materials and is never observed in portland cements or limes.*
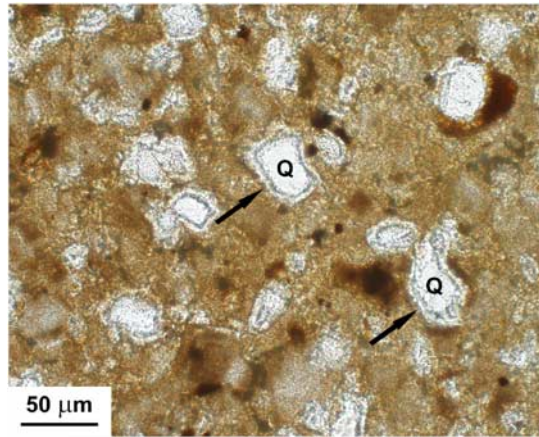
FIG. 11—*The cement shown in this plane polarized light photomicrograph was created in the laboratory by burning the lower member of the Rondout Formation rocks at 1000°C for 24 h. The original quartz grains (Q) have begun to react either forming trydimite (a different structural form of quartz) or have combined with the surrounding carbonates to form a calcium silicate. Even at this temperature, a large degree of the original raw feed texture is preserved. The arrows indicate the thin rims formed around the quartz grains as a result of the burning process.*

the superficial similarities. Alite with its high refractive index will appear to stand out above the plane of the thin section while quartz will appear "flat" against its surroundings. The colors produced by the polarized light interference are subtly different but significant enough to detect by the trained eye. Alite will appear a dull gray while quartz may appear gray to white. When accurate optical properties are collected there should be no mistaking the difference. Even so, the preservationist should be wary if alite is identified in a report generated for a structure built before approximately 1880. While European portland cements were available, natural cement is the much more likely cementitious component.

Discussion

Good forensic laboratory practices can go a long way toward revealing the materials comprising historical construction. Petrographic techniques that are often as old as the materials being studied can play a lead role in the analysis. Reasonably accurate reverse engineering of cementitious materials is quite possible when microscopy techniques are combined with chemical analysis. For the sake of clarity, the most
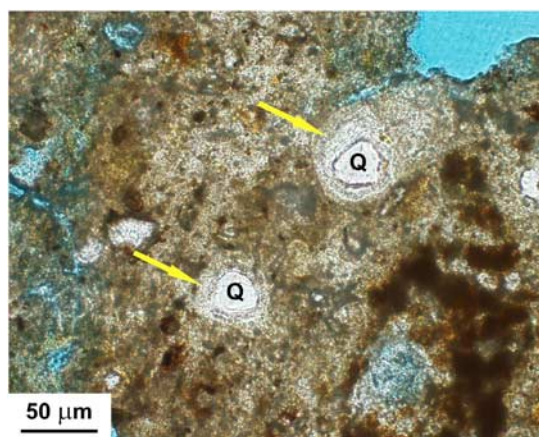

FIG. 12—*Depicted here in this plane polarized light photmicrograph is a mortar produced with natural cement. The arrows indicate reaction rims that formed around quartz grains (Q) during the burning process. These textures may be confused with hydration rims surrounding alite in portland cement (see Fig. 5). However, the optical properties are distinctly different and careful petrographic examination should preclude any confusion.*

distinctive petrographic features are described in this article. It should be understood that when multiple binders are present or when significant deterioration has occurred, the analysis may become substantially more complicated.

If nothing else, the article should have dispelled the black box myth. The results of a mortar analysis report are only as good as the sample provided, the techniques applied by the analyst, and the investigator's experience. If material identification and replacement-in-kind are of importance then the laboratory analysis should be given high consideration.

Only a small portion of a typical mortar analysis has been discussed. Completely neglected is the aggregate analysis performed by a complete dissolution of the binder matrix and petrographic description of the intact residues. Chemical analysis and the several accompanying destructive procedures have only been summarily addressed. Consider the search for large binder residuals and the preparation of large area thin sections for petrography and the need for significant sample becomes obvious. Powder scrapings from the mortar joint will reveal little to nothing. Consider providing a full bed joint for brick construction or a significant portion thereof for stone masonry. Samples collected away from atmospheric or environmental exposure better represent the original material.

The laboratory should be considered an important member of the preservation team. Retaining a laboratory that performs only routine examinations and has no real experience in historical materials analysis is equivalent to hiring a masonry contractor that only repoints Type N portland-lime mortars and has no experience in placing restoration materials. A brief discussion of the project with the laboratory manager will often reveal volumes about the skill of that laboratory in reverse engineering historical materials. When an experienced laboratory is found, the forensic work should be performed as early as possible as the analyses described above will require several weeks for completion.

It is refreshing that a momentum is gathering toward an appreciation for authentic American masonry materials and practices. Many decades of inappropriate restorations have been suffered with the application of portland cement-based repair materials "softened" with hydrated lime. The recent interest in hydraulic limes as a restoration alternative represents a quantum leap in sophistication. Where natural cement beds are present, limes are much more physically compatible than portland cement mortars and further deterioration will likely be avoided through their application. However, where true replacement-in-kind is desired, limes are as inaccurate as portland cement when natural cements are present in the construction. Foremost in a determination of an appropriate repair material is an identification of the original binders and their proportions. It is hoped this paper will spark further interest in the forensic aspects of American masonry construction and that the scientific data will keep pace with the increasing desire among preservation professionals for accurate analysis of existing materials.

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