Laboratory Analyses Of Masonry Mortars From Fort Washington, Maryland

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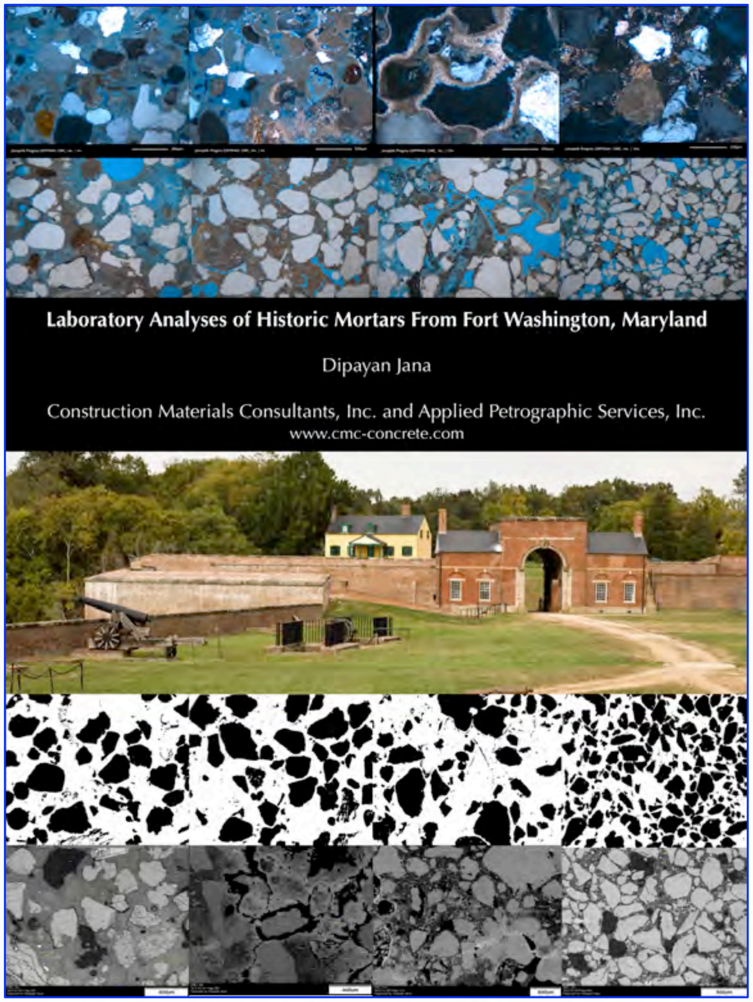
Both the Professional Report & the Presentation can be downloaded from the publications page of our website at www.cmc-concrete.com

Professional Report



Presentation

Why Do We Need Laboratory Analysis Of Masonry Mortar?

Mortar Sands
- ✦ Natural vs. Manufactured Sand
- ✦ Siliceous Sand
- ✦ Calcareous Sand
- ✦ Mixed Sand
- ✦ Sea Shells
- ✦ Ceramics

Mortar Binders
- ✸ High-Calcium (Non-Hydraulic) Lime
- ✸ Magnesian or Dolomitic Lime
- ✸ Hydraulic Lime (Natural Hydraulic Limes)
- ✸ Natural cement
- ✸ Portland Cement
- ✸ Blended Cement
- ✸ Slag Cement
- ✸ Masonry Cement

Mortar Types
- ✧ Lime Mortar
- ✧ High-Calcium vs. Dolomitic Lime Mortar
- ✧ Non-hydraulic vs. Hydraulic Lime Mortar
- ✧ Natural Cement – Lime Mortar
- ✧ Cement-Lime Mortar
- ✧ Masonry Cement Mortar

Mix Proportions
- ★ Lime-Sand Ratio (Lime Content, Sand Content)
- ★ Cement-Lime-Sand Ratio (Lime-Cement-Sand Contents)
- ★ Improper Mix – Over-sanded, Under-sanded, Improper lime-to-cement ratio for a masonry unit

Pigments, Fillers, Pozzolans
- ⦿ Pigments (Carbon, Mineral Oxides)
- ⦿ Fillers (Limestone Fines, Quartz Fines, Ceramic Dusts)
- ⦿ Pozzolans (Natural vs. Manufactured)

Mortar Deteriorations
- ☑ Shrinkage
- ☑ Expansion
- ☑ Softening, Dusting
- ☑ Lime Leaching
- ☑ Secondary Calcite Precipitation
- ☑ Secondary Gypsum From Acid Rain
- ☑ Efflorescence
- ☑ Salt-related Distress

Acid Digestion – Mason's Favorite Test



Sand color & grain-size distribution

Assumption
Binder entirely dissolves in acid

Problem
- Gypsum, Clay, Pigments
- Hydraulic Component, Pozzolans
- Multiple Binders
- Leaching, Alterations

Residue = Sand Content (lb.)

Mortar weight minus Sand weight

Dissolved = Binder Content (lb.)

Binder (bulk density 40 lbs/ft^3)

Sand (bulk density 80 lbs/ft^3)

Binder : Sand, by volume

Assumption
Sand does not dissolve in acid

Problem
- Calcareous sand
- Marine shells
- Beach sands
- Mixed siliceous-calcareous sand
- Soluble constituents in silica sand

Acid vs. Water Digestion – Mortar From The Fort Wall

Acid Digestion

Sand from Acid-Insoluble Residue = 68.6%
Binder = 31.4%

Sand Bulk Density = 80 lbs./ft^3
Binder Bulk Density = 40 lbs./ft^3

Sand Volume = 0.858
Binder Volume = 0.785

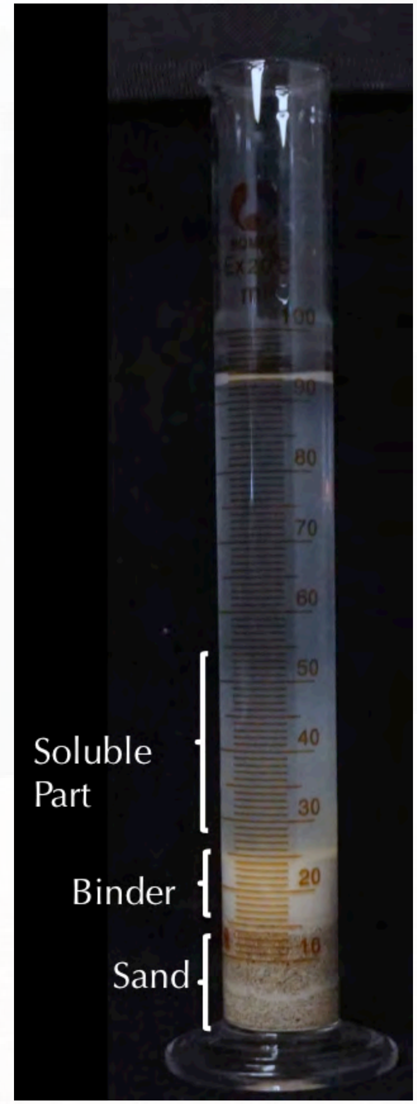
Binder : Sand = 1:1.1

Grossly underestimated sand

Water Digestion

Binder : Sand = 1:1.6

Underestimated sand



Laboratory Analysis of Masonry Mortar

1. Optical Microscopy

- Sand Type
- Sand Size Distribution & Color Variations
- Binder Type(s), Raw Feed
- Mortar Types
- Texture & Microstructure
- Mix Proportions
- Calcareous Sand, Sea Shells
- Pozzolans, Slag, Filler, Pigment
- Mortar Deteriorations, Alterations

2. Scanning Electron Microscopy & X-ray Microanalysis

- Binder Types From Paste Chemistry
- (Lime, Hydraulic Lime, Natural Cement, Portland Cement Binders from CaO-MgO-SiO_2 contents of Paste
- Hydraulicity from CI of Paste
- Mortar Types
- Calcareous Sand, Sea Shells
- Pozzolans, Slag, Filler, Pigment
- Mortar Deteriorations

3. Acid Digestion (Wet Chemical)

- Binder to Sand Ratio from
- Acid-Insoluble Residue (Siliceous Sand) Content
- Sand Size Distribution & Color Variation (Siliceous Sand)
- Filtrates for Soluble Silica, XRF, AAS, ICP (Dissolved Binder)

Problems with Acid Digestion

- Calcareous Sand, Sea Shells
- Pigments, Pozzolans, Slag, Clay, Gypsum
- Leaching and Alterations

4. Gravimetry

- Loss on Ignition at
- 110°C (Free Moisture Content)
- 550°C (Combined Water Content)
- 950°C (Carbonates and Carbonation)

Problems with Gravimetry

- Calcareous Sand, Sea Shells
- Pigments, Pozzolans, Slag, Leaching

5. Instrumental Chemical Analysis

(XRF, AAS, ICP)

- Bulk Composition
- Binder Composition
- Soluble Silica From Binder

6. X-ray Diffraction

- Sand Mineralogy
- Binder Mineralogy
- Deleterious Constituents
- Efflorescence and Other Salts
- Pigments, Additives, Fillers

7.Thermal Analysis

(DTA, TGA, DTG, DSC)

- Binder and Mortar Type
- Hydrates, Sulfates, and Carbonates
- Dolomitic Lime Content from Brucite
- Deleterious Constituents, Salts
- Quantitative Analysis

8. Ion Chromatography

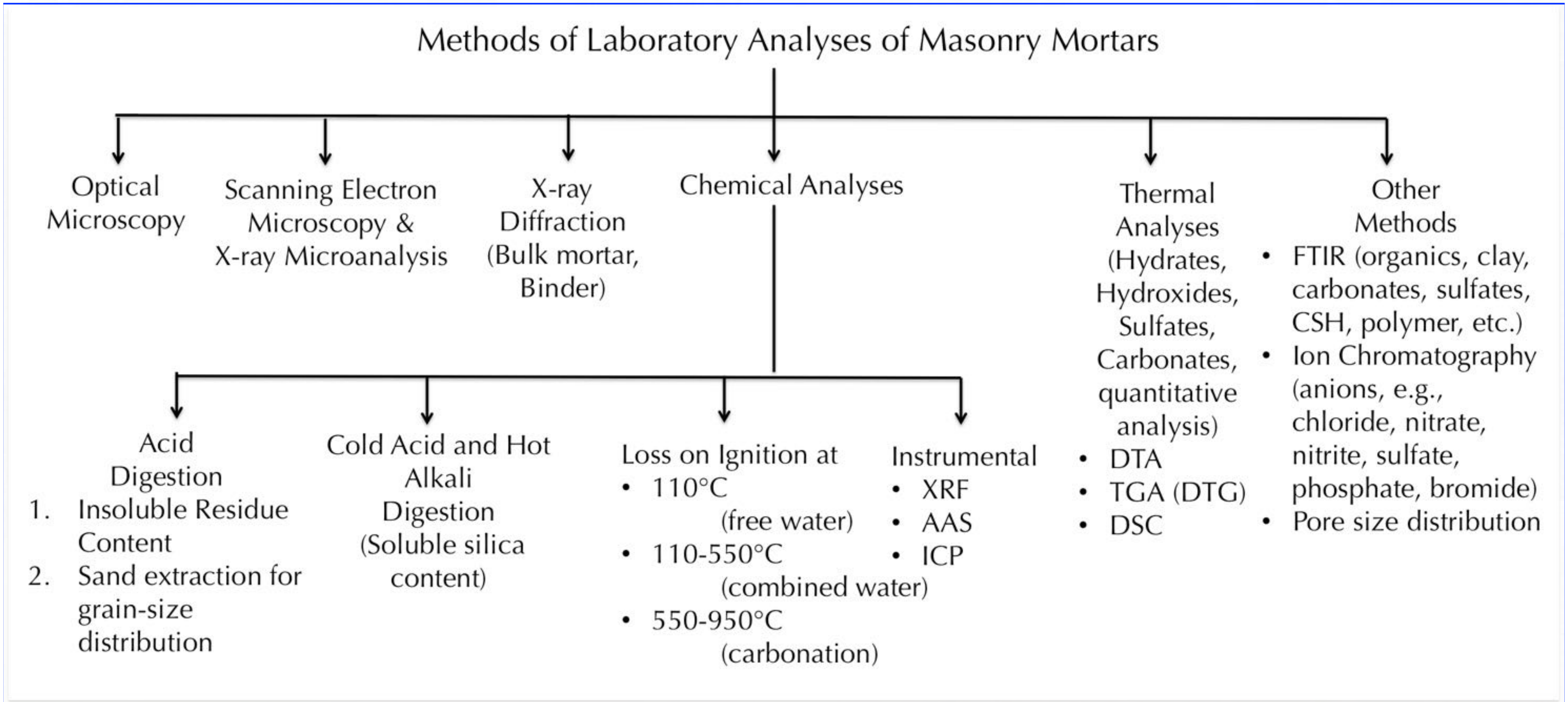
- Soluble Salts (e.g., Cl^-, SO_4^{2-}, NO_3^-)

9. Infrared Spectroscopy

- Organic compounds
- CSH, Sulfate, Carbonate, Hydrates

Sequence of steps to be followed in laboratory analysis

Laboratory Analysis of Masonry Mortar



Sequence of steps to be followed in laboratory analysis

Test Methods on Analysis of Masonry Mortar – ASTM C 1324, RILEM (Middendorf et al. 2005)

Designation: C 1324 – 96

Standard Test Method for Examination and Analysis of Hardened Masonry Mortar[1]

Designation: C1713 – 15

Standard Specification for Mortars for the Repair of Historic Masonry[1]

ASTM C 1324: Laboratory analysis of hardened mortar

- Petrographic Examinations (Optical Microscopy, SEM)
- Chemical Analyses
  (Insoluble Residue, Soluble Silica, Loss on Ignition)
- XRD
- Thermal Analysis

ASTM C 1713: Mortars for the Repair of Historic Masonry

RILEM: Investigative methods for the characterization of historic mortars

- Part I: Mineralogical Characterization
- Part II: Chemical Characterization

Available online at www.rilem.net

Materials and Structures 38 (October 2005) 761-769

RILEM TC 167-COM: 'Characterisation of Old Mortars with Respect to their Repair'

Investigative methods for the characterisation of historic mortars – Part 1: Mineralogical characterisation

Available online at www.rilem.net

Materials and Structures 38 (October 2005) 771-780

RILEM TC 167-COM: 'Characterisation of Old Mortars with Respect to their Repair'

Investigative methods for the characterisation of historic mortars - Part 2: Chemical characterisation

Laboratory Analysis – Proper Sampling is the Key

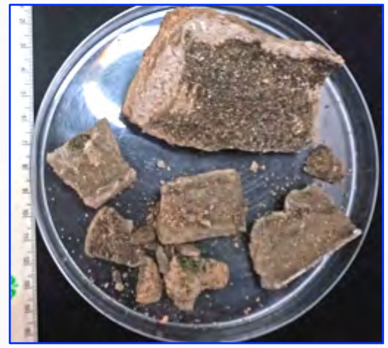
1. Take a photo of the location from where mortar sample will be taken

2. Take a photo of the joint from where mortar sample will be taken

3. Use a flat head chisel and hammer to carefully remove the jointing mortar as intact as possible

4. Mark the Sample ID and exposed side on the sample

Please be liberal in sampling



- Provide at least 50 grams to 100 grams of sample in a sealed Ziploc bag
- Preferably multiple intact pieces, not powders
- Of uniform appearance
- Representative of the purpose of examination

- Avoid providing multiple sample types, e.g., brick chips mixed with mortar, or original mortar mixed with later repointing mortar

- From multiple intact pieces adequate representative samples can be selected for microscopy, chemical, XRD-XRF, sand size distribution, and other laboratory tests

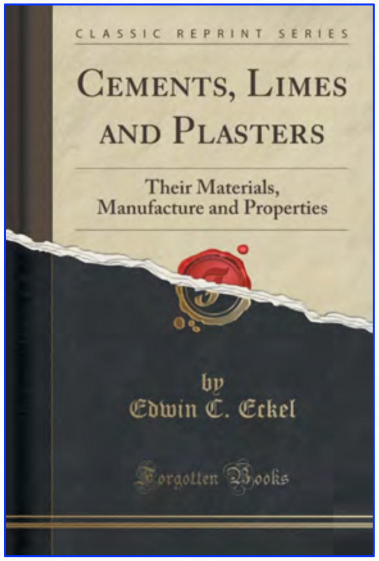
Mortar Samples From Fort Washington

25g 15g 26g 260g 58g 208g 1151g

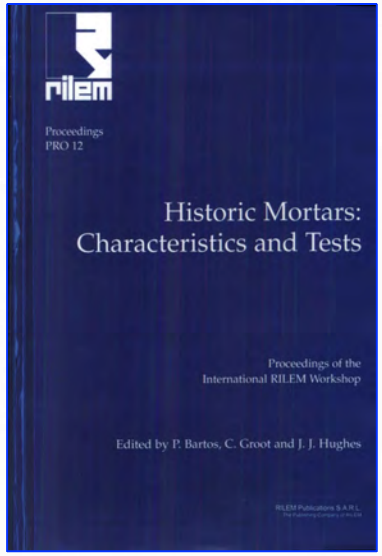
Publications on Historic Mortar Analysis

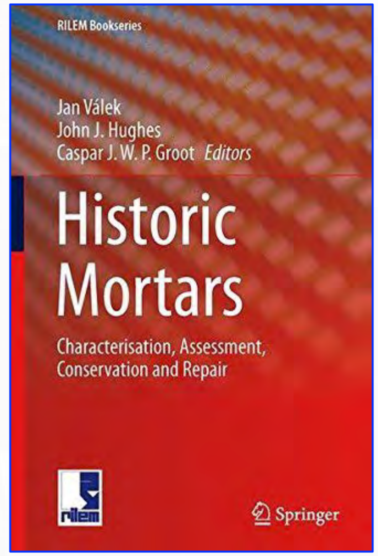
A Masterpiece!

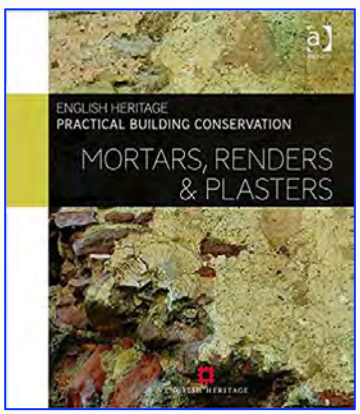
Eckel 1922

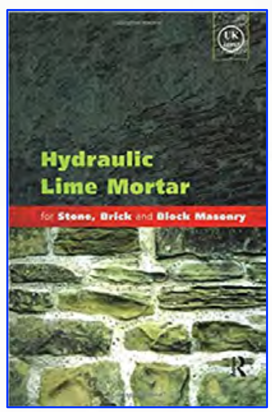


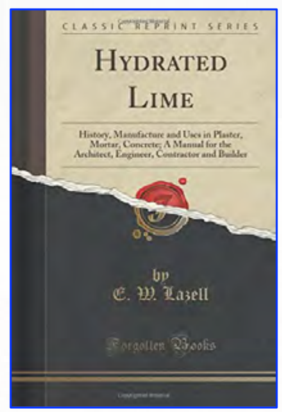
Three Excellent Resources on Laboratory Testing of Historic Mortars

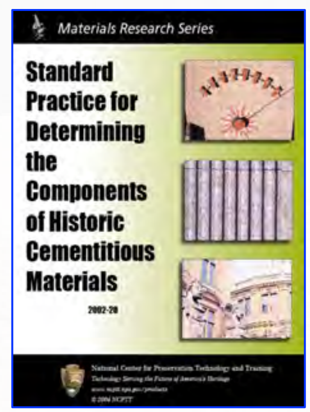


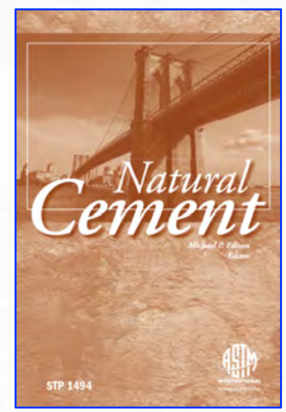




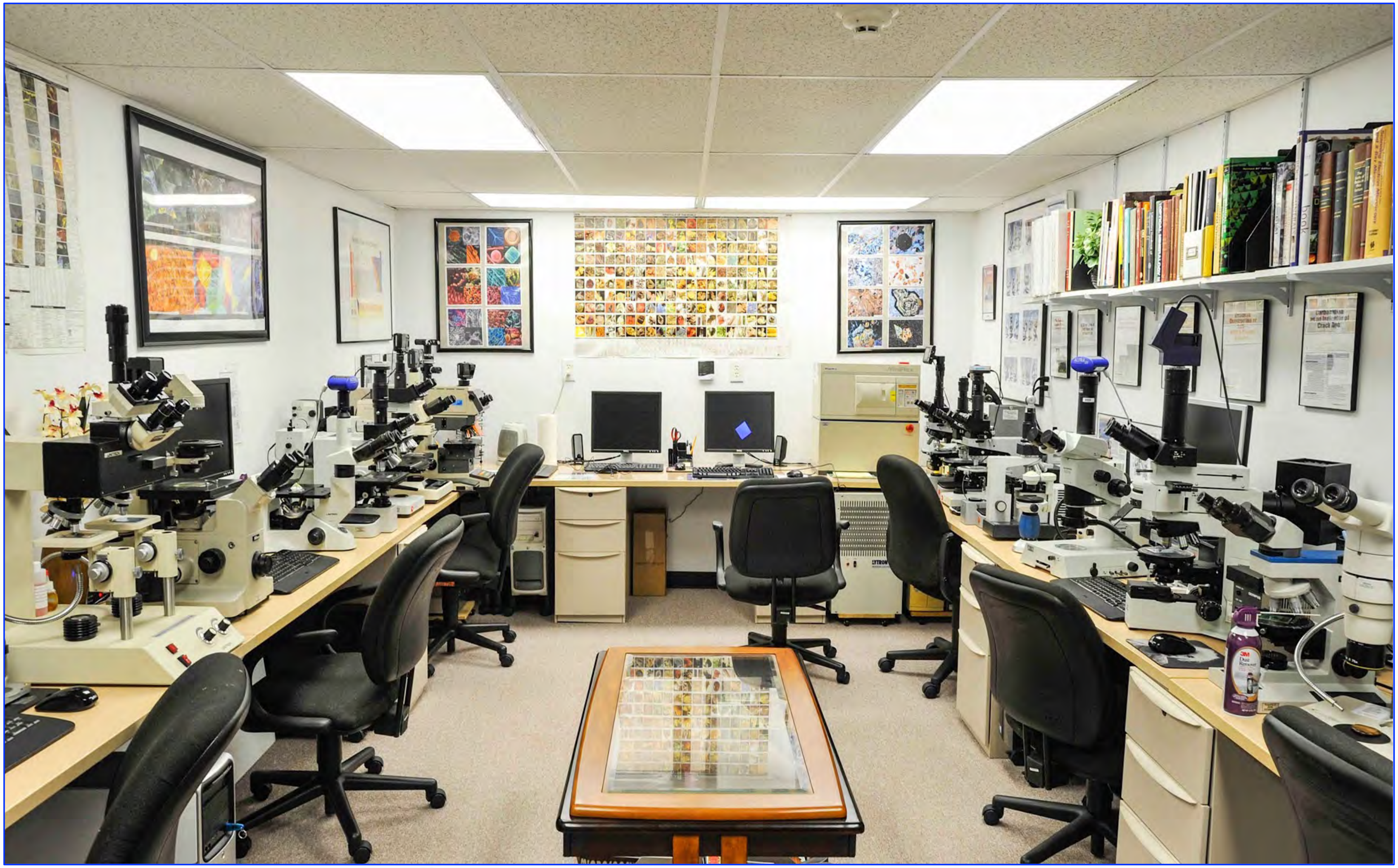








Optical Microscopy



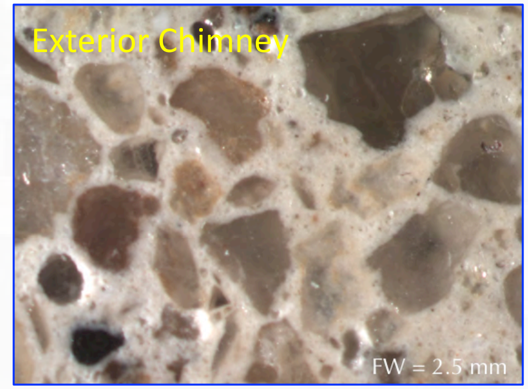
Three Essential Optical Microscopes for Mortar Analysis

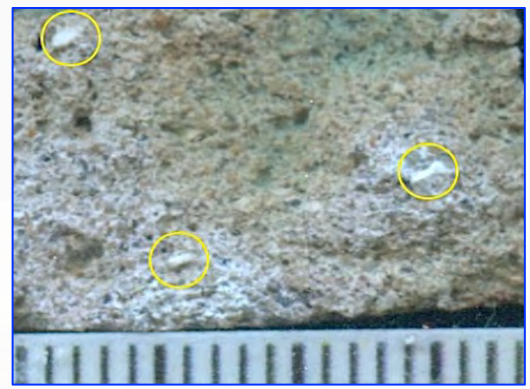
Nikon Eclipse E600 POL Polarizing(Petrographic) Microscope

Olympus SZH Reflected/Transmitted/Polarized Stereozoom Microscope

Nikon SMZ-10A Stereozoom Microscope

Low-power Stereo Microscope for Mortar Analysis

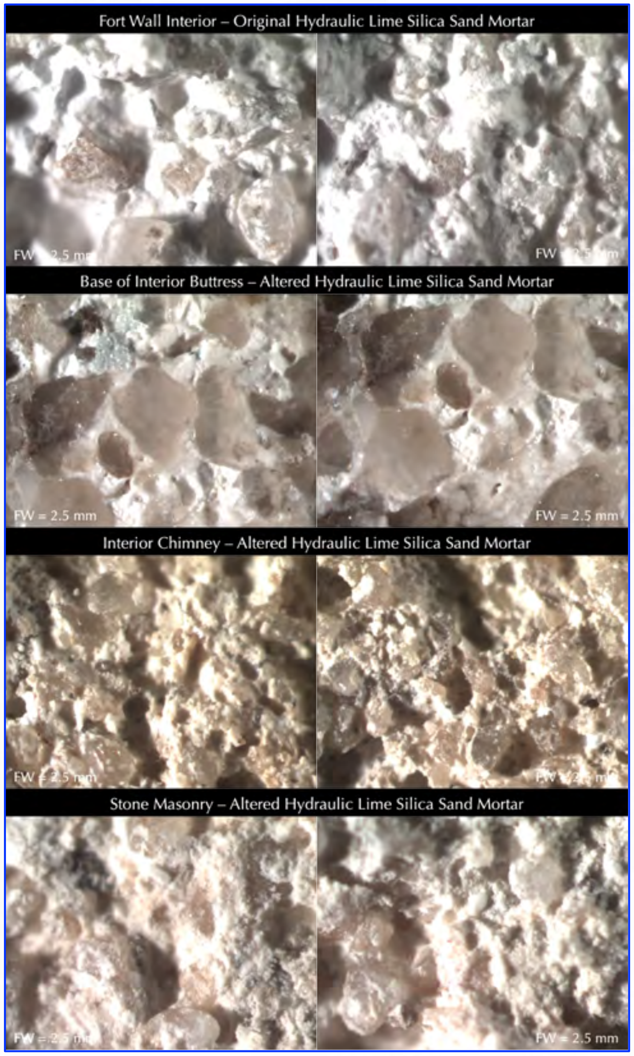


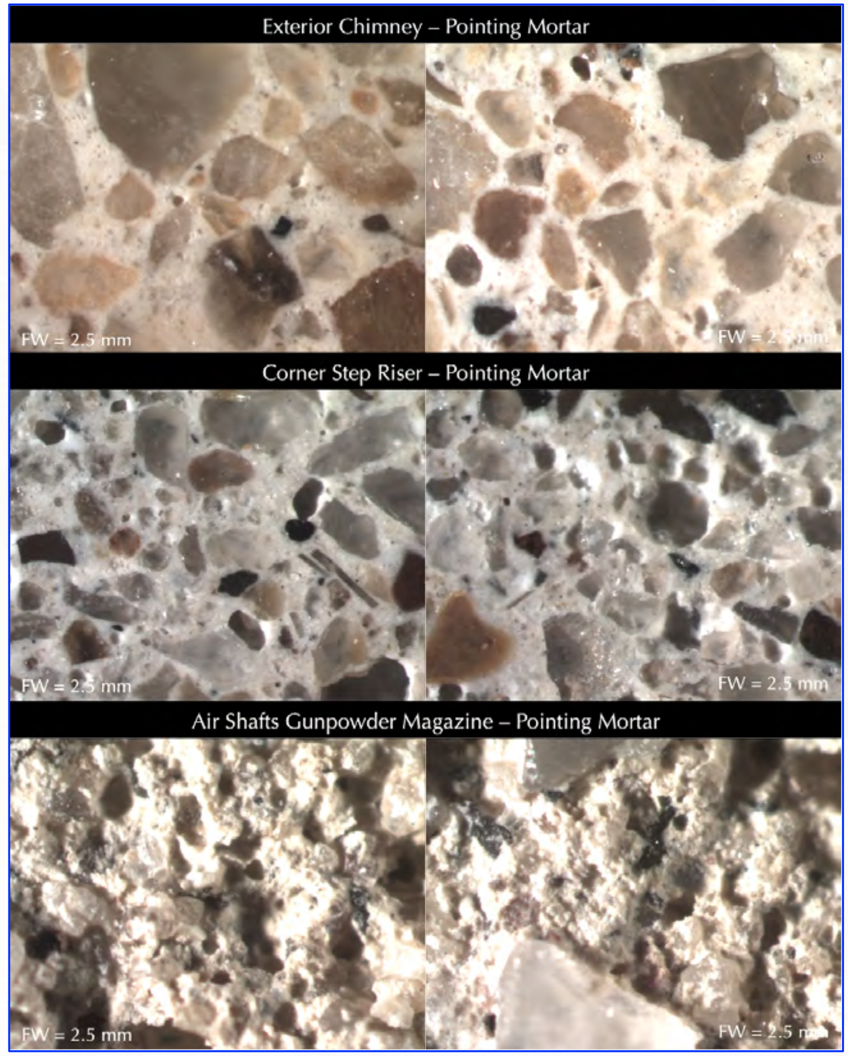


Sand types, color, size, shape, angularity, gradation, paste color and hardness, lime lumps, air entrainment

Low-power Stereo Microscope for Mortar Analysis

Original Lime Mortars

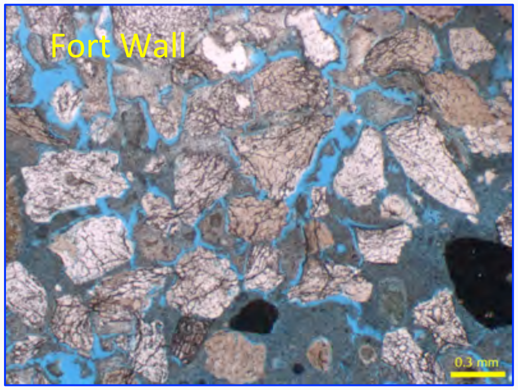


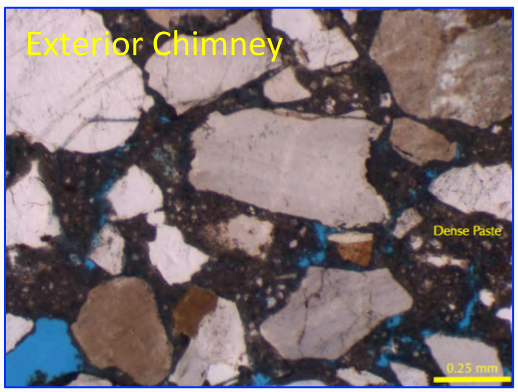


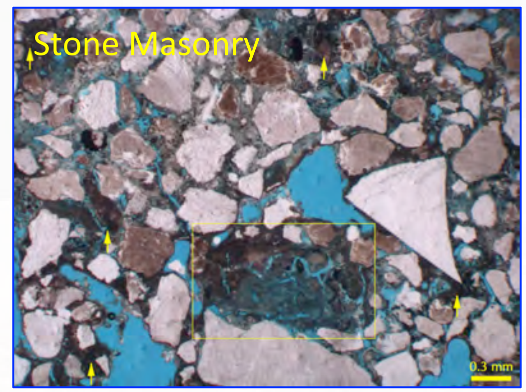
Later Cement - Lime Mortars

Sand types, color, size, shape, angularity, gradation, paste color and hardness, air entrainment

Transmitted and Polarized-Light Stereo Zoom Microscope for Mortar Analysis

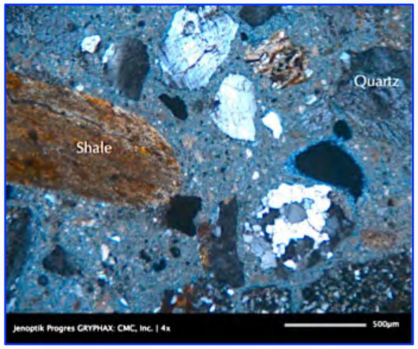




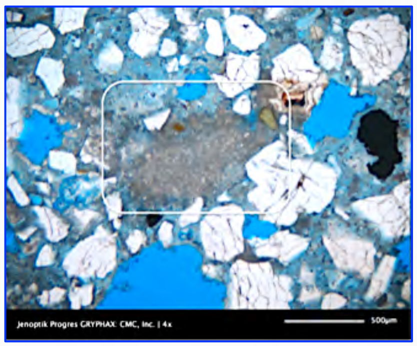


Dense vs. porous paste, carbonated paste, shrinkage microcracks, sand type, lime lumps, air entrainment

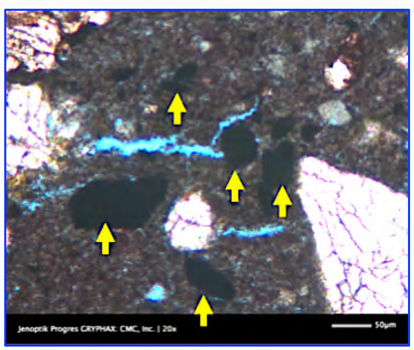
Petrographic Microscope for Mortar Analysis



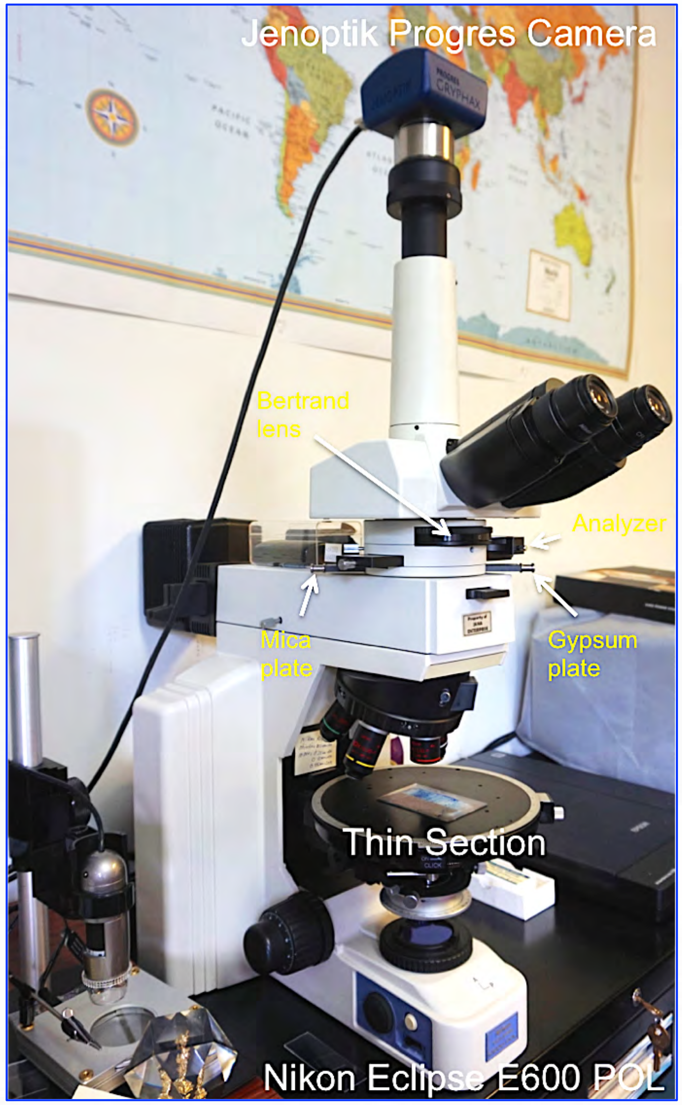
Sand Mineralogy, Texture, Type, Soundness



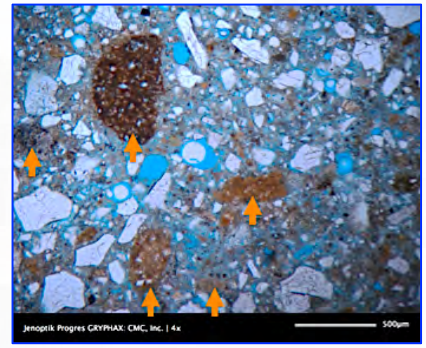
Dolomitic Hydraulic Lime Mortar



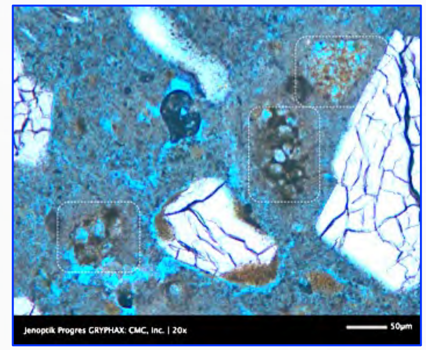
Pigmented Lime Mortar



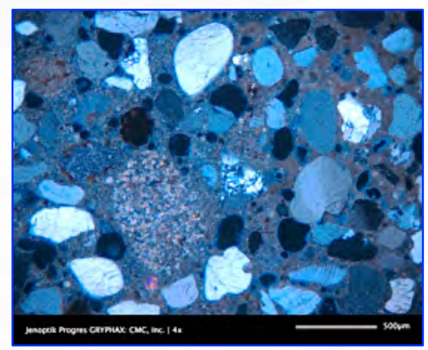
Petrographic Microscope – Powerhouse of Mortar Analysis



Natural cement - Lime Mortar



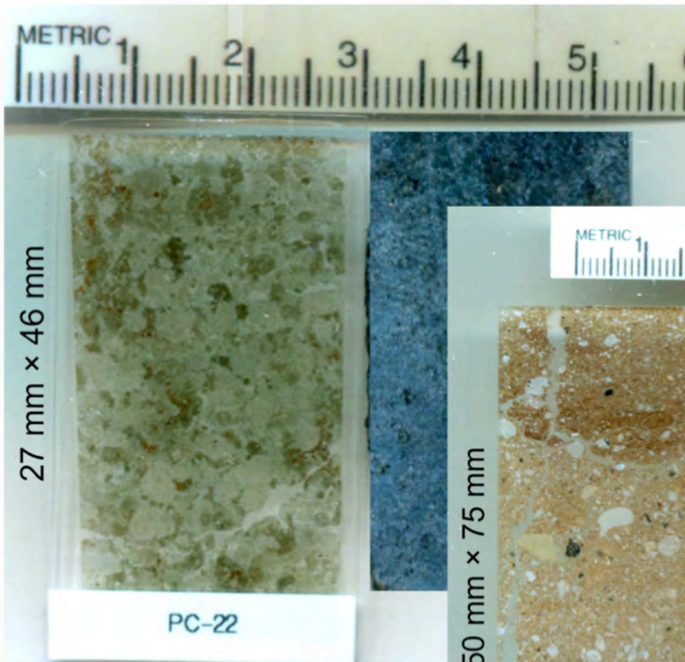
Portland cement - Lime Mortar



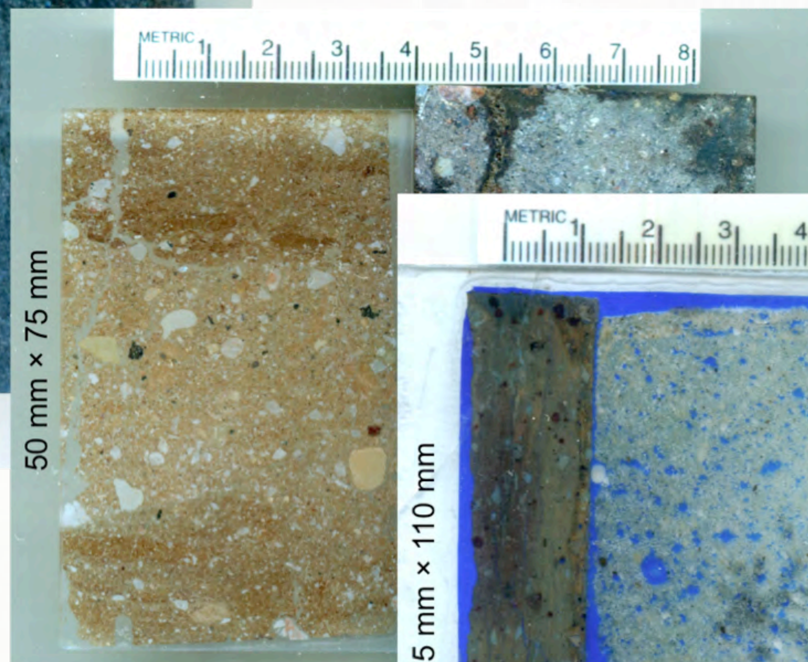
Masonry cement Mortar

Thin Section Microscopy – At the Heart of Mortar Analysis

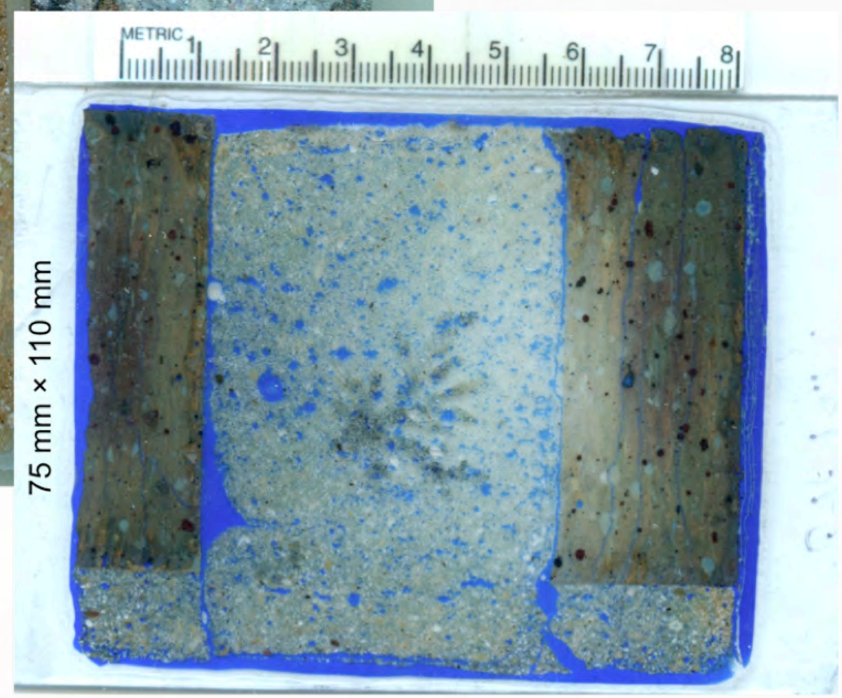
Impregnation
- No Epoxy
- Clear Epoxy
- Colored Epoxy
- Fluorescent Epoxy



Rocks

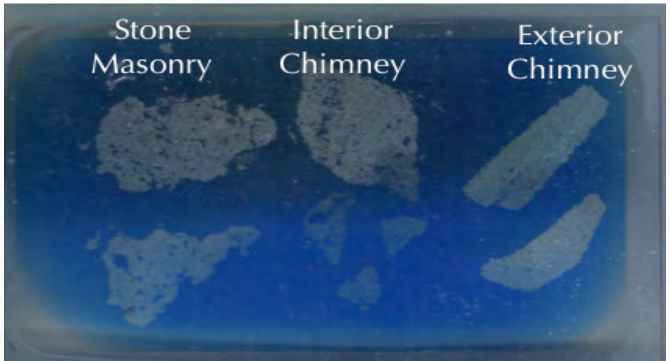


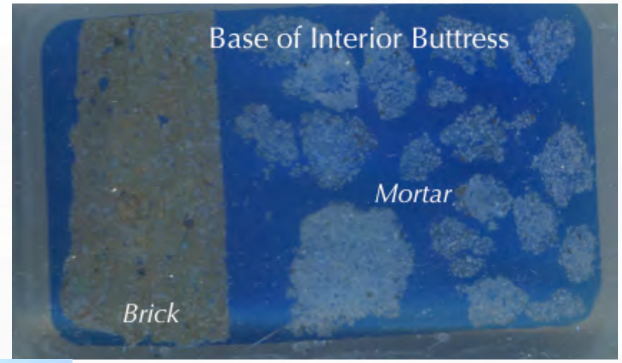
Concrete



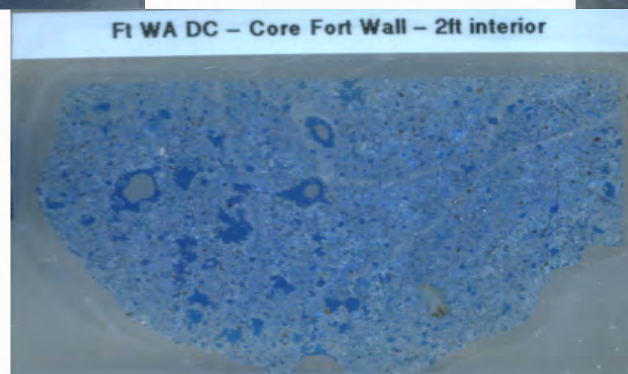
Masonry

Thin Section Microscopy – At the Heart of Mortar Analysis

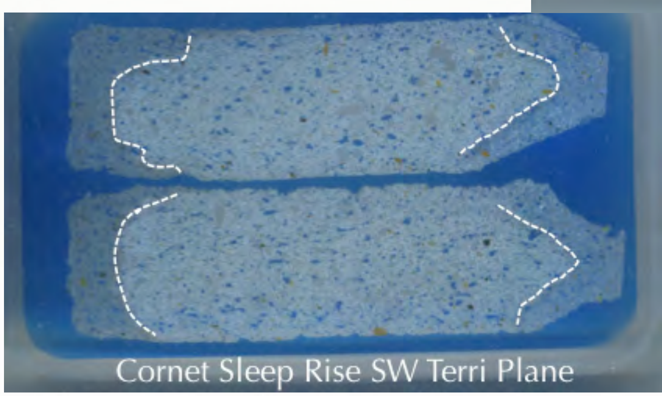


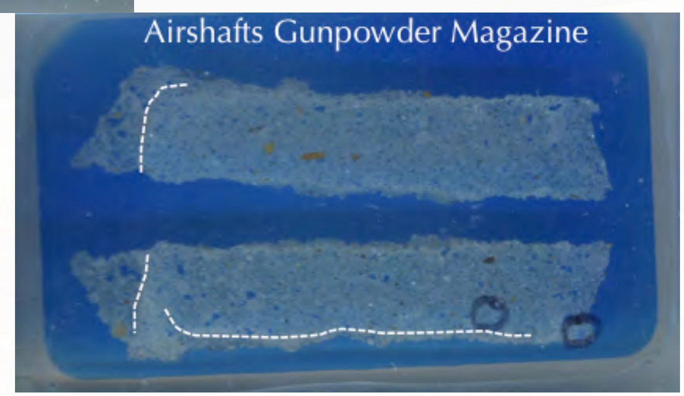


50 mm × 75 mm size blue dye-mixed epoxy-impregnated thin sections

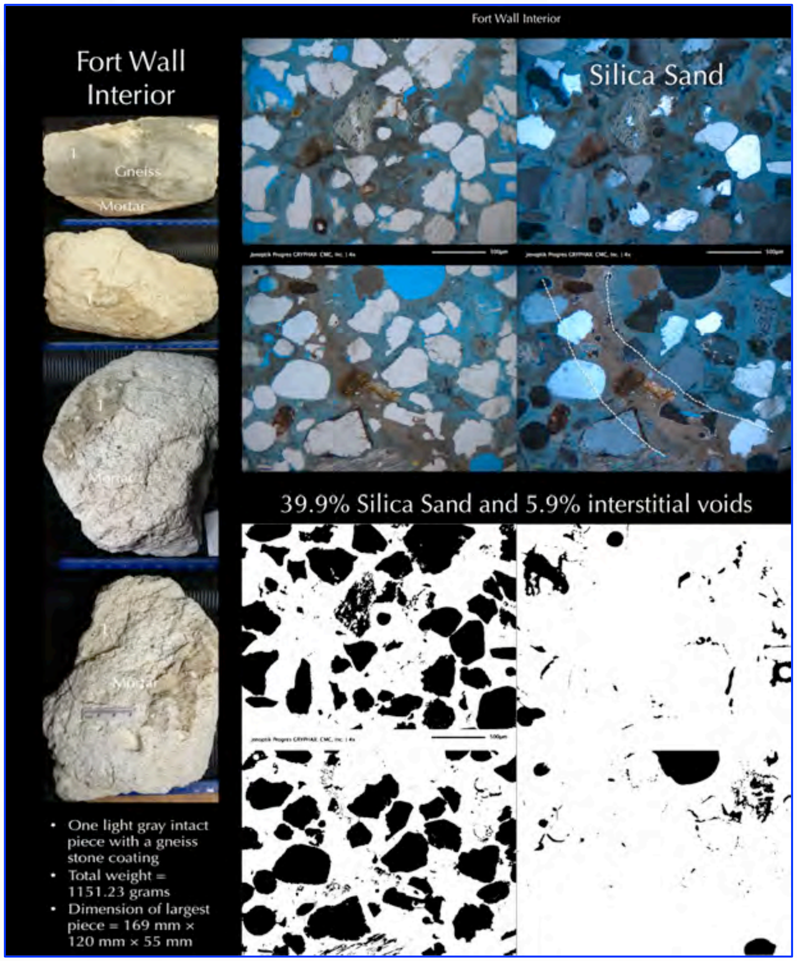


Blue dye highlights pore and void spaces, cracks, and porous areas of paste

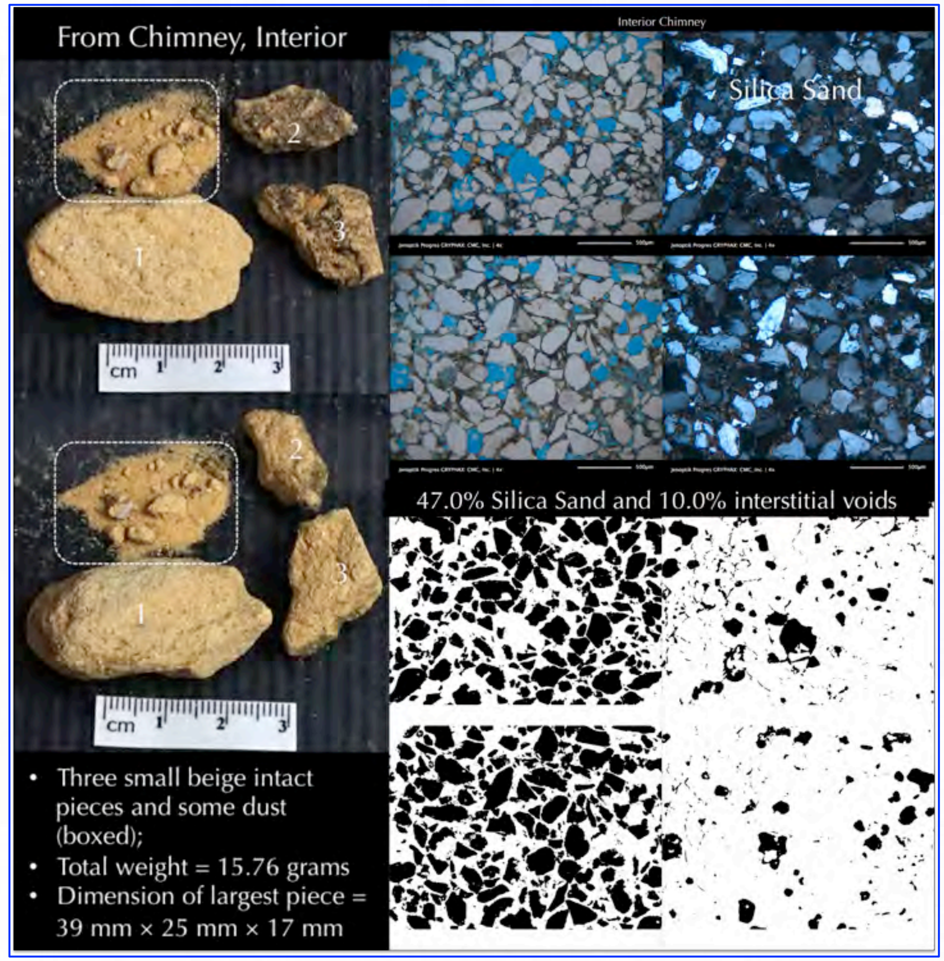




Optical Microscopy & Image Analysis

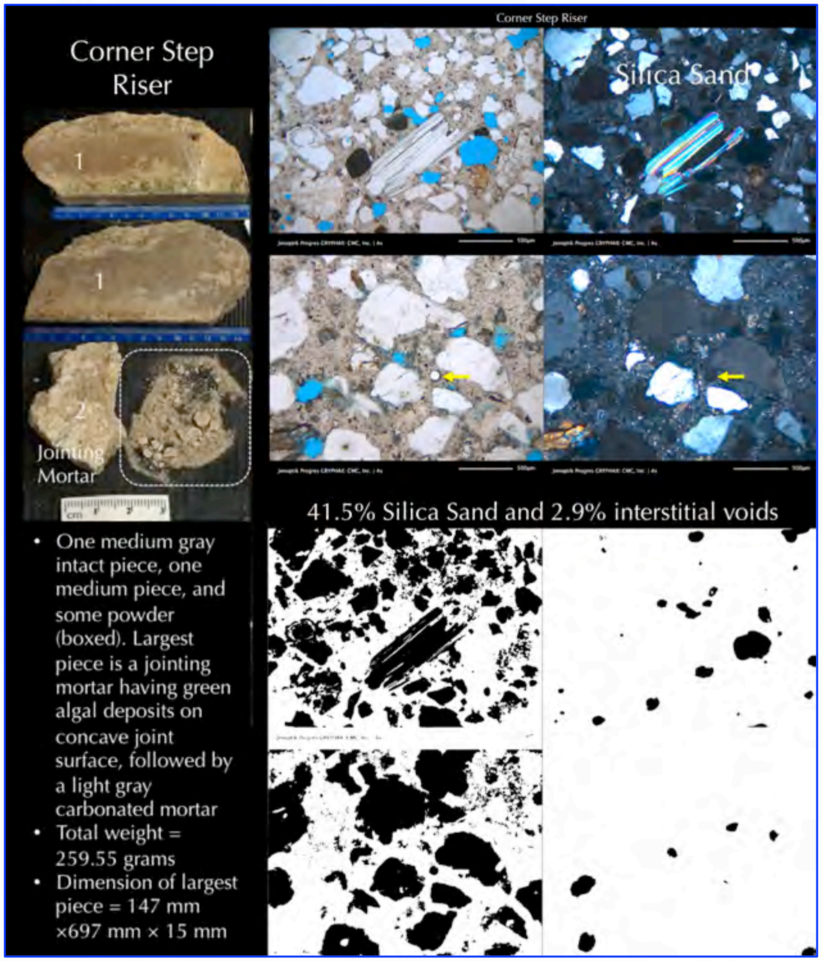


Sand in most samples from the Fort as in mortar from interior of Fort Wall

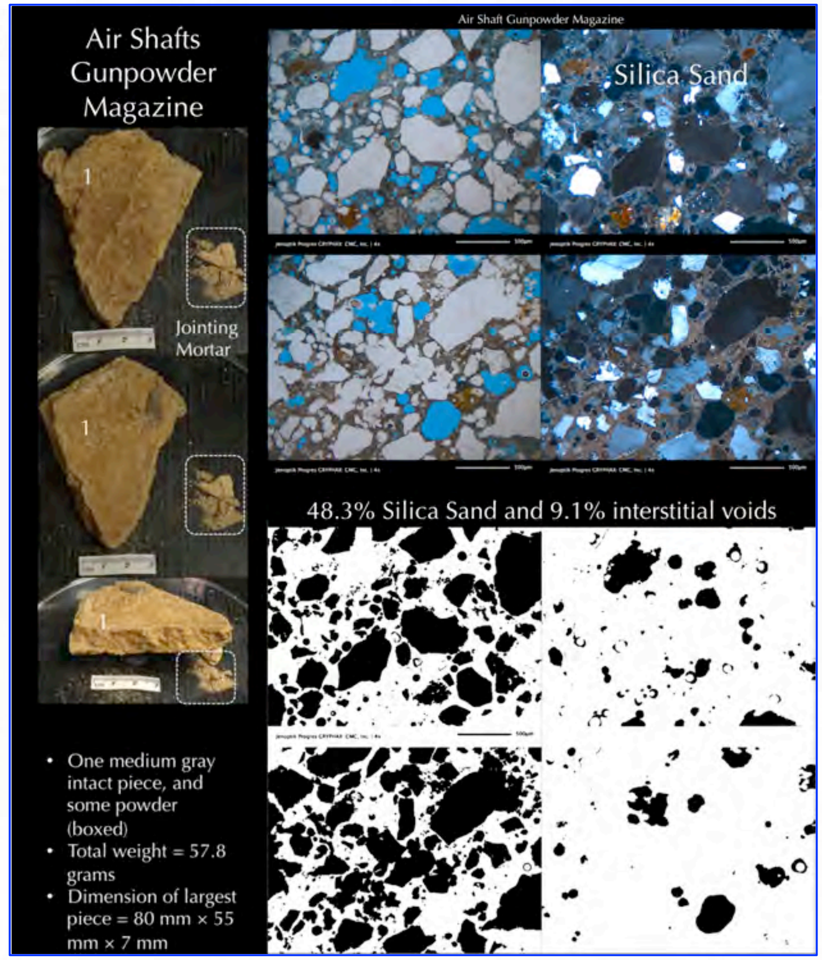


Finer, crushed sand in mortar from Interior Chimney

Optical Microscopy & Image Analysis

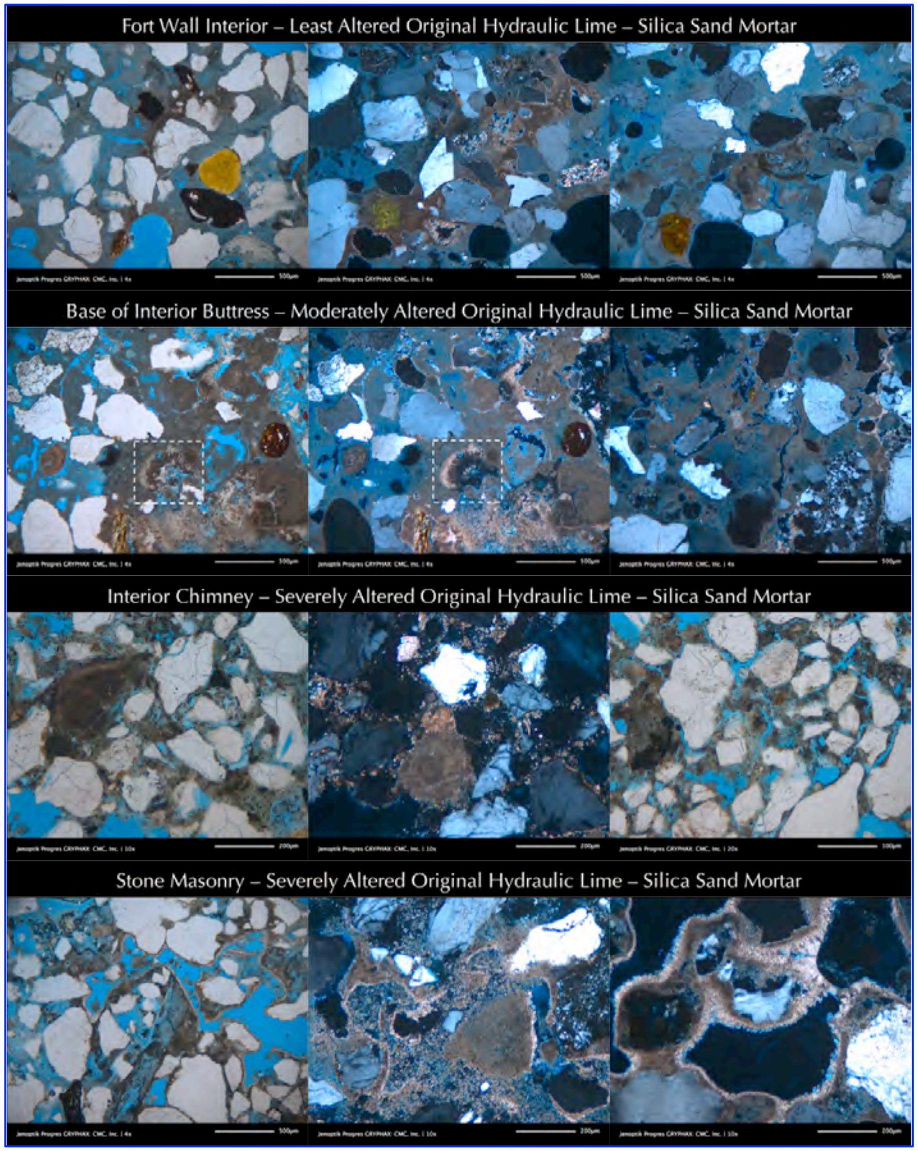


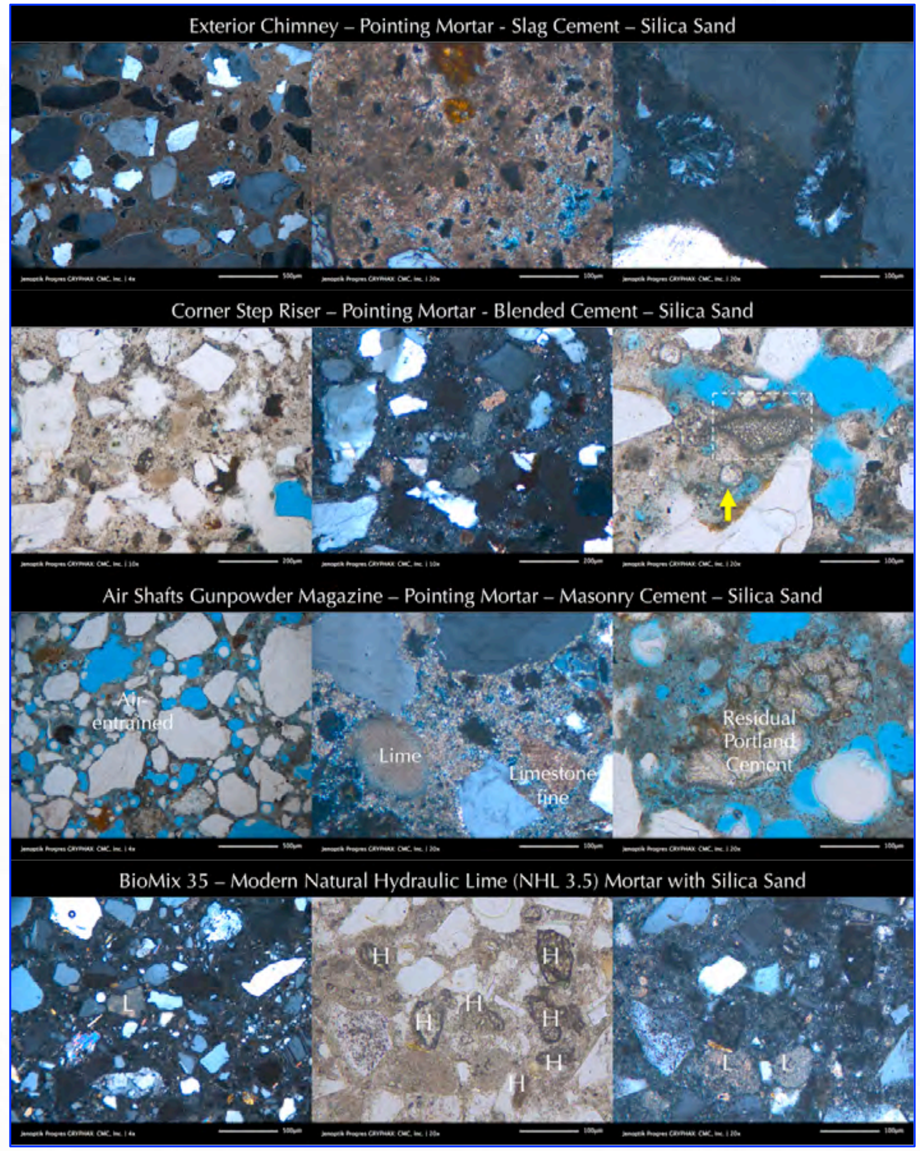
Dense, non-air-entrained Corner Step Riser Terri Plane mortar



Air entrainment from masonry cement in Air Shafts Gunpowder Magazine

Optical Microscopy

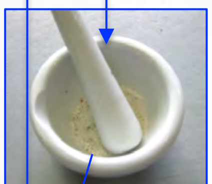


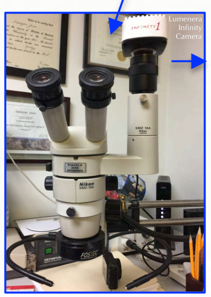


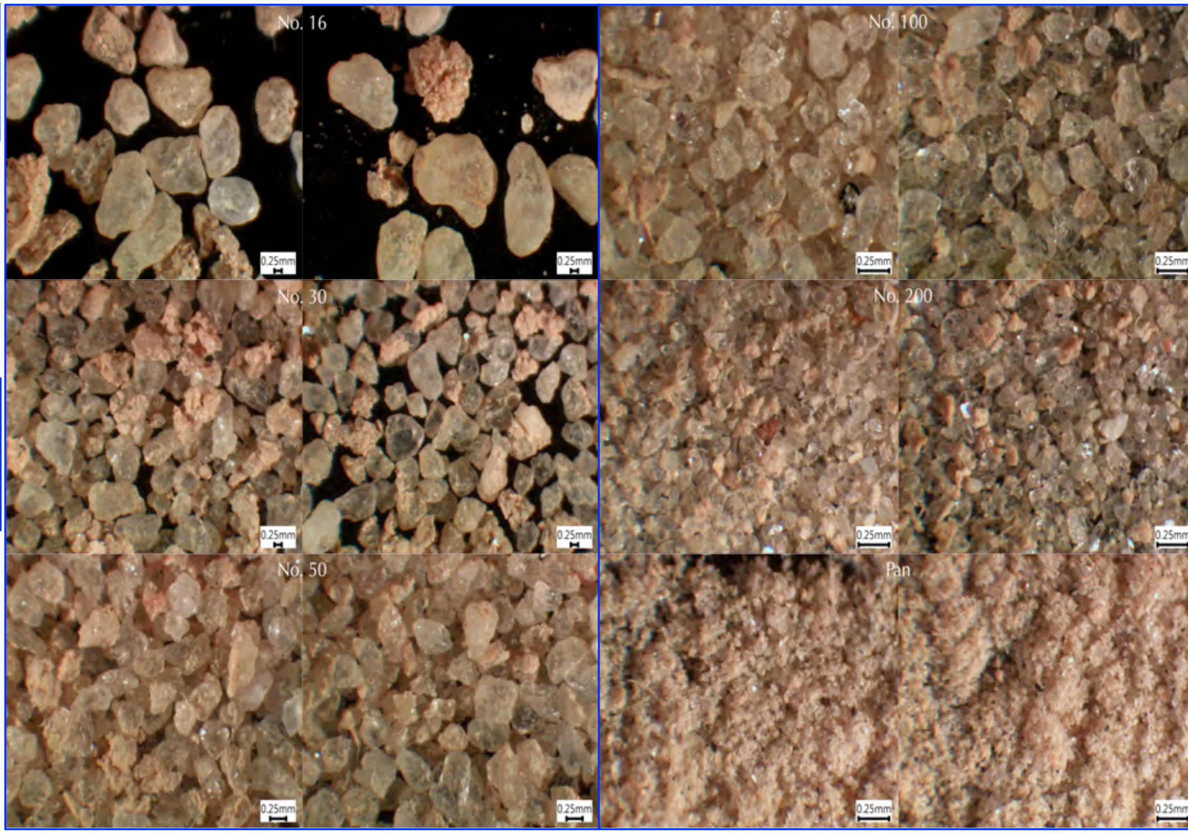
Sand Extraction, Size Distribution & Color Variation

Sand extraction by acid digestion with minimal or without crushing followed by Sieve Analysis in Gilson Mini Sieve Shaker and examination in a stereo microscope

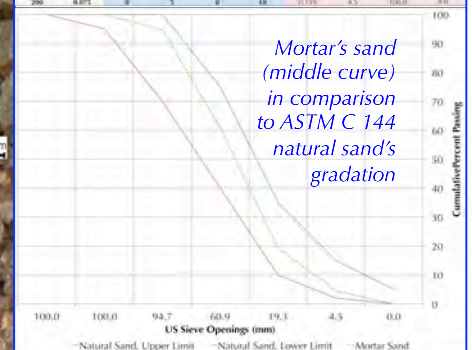
Photomicrographs of sand retained on various sieves

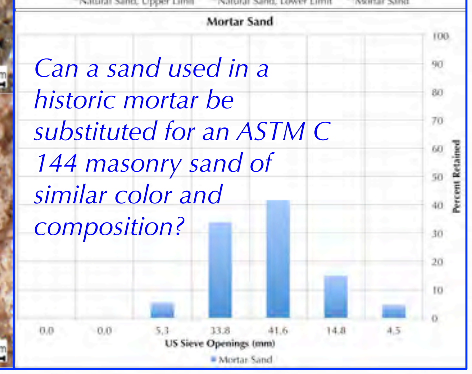






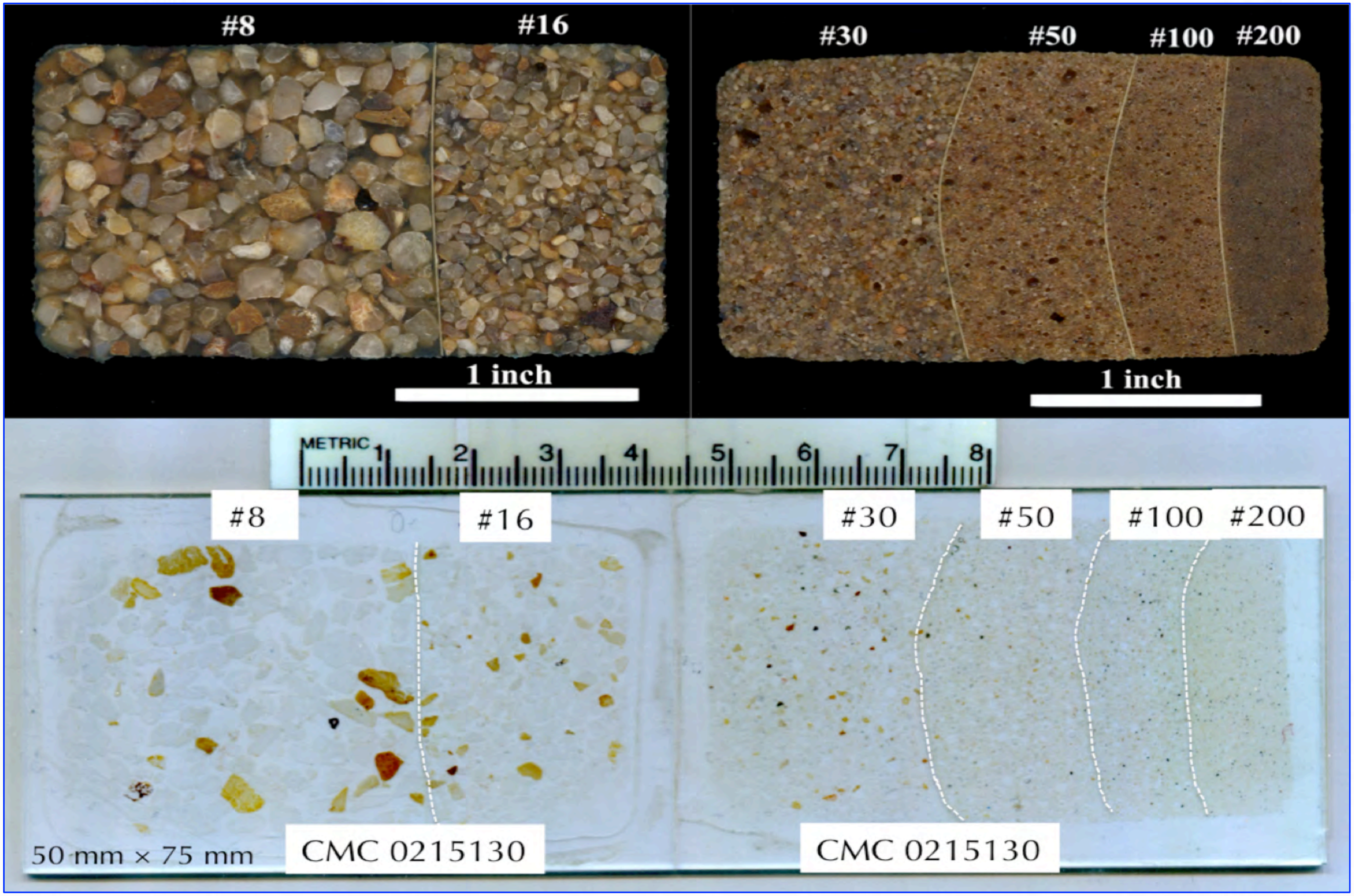
Sand Sieve Sizes		Percent Passing for Natural & Manufactured Sand				Weight Retained, g	Percent Retained	Cumulative Percent Retained	Percent Passing
US Sieve No.	mm	Natural Sand Lower Limit	Natural Sand Upper Limit	Manufactured Sand Lower Limit	Manufactured Sand Upper Limit	Mortar	Mortar	Mortar	Mortar
4	4.75	100	100	100	100	0.000	0.0	0.0	100.0
8	2.36	95	100	95	100	0.000	0.0	0.0	100.0
16	1.18	70	100	70	100	0.186	5.3	5.3	94.7
30	0.6	40	75	40	75	1.190	33.8	39.1	60.9
50	0.3	10	35	20	40	1.463	41.6	80.7	19.3
100	0.15	2	15	10	25	0.522	14.8	95.5	4.5
200	0.075	0	5	0	10	0.159	4.5	100.0	0.0



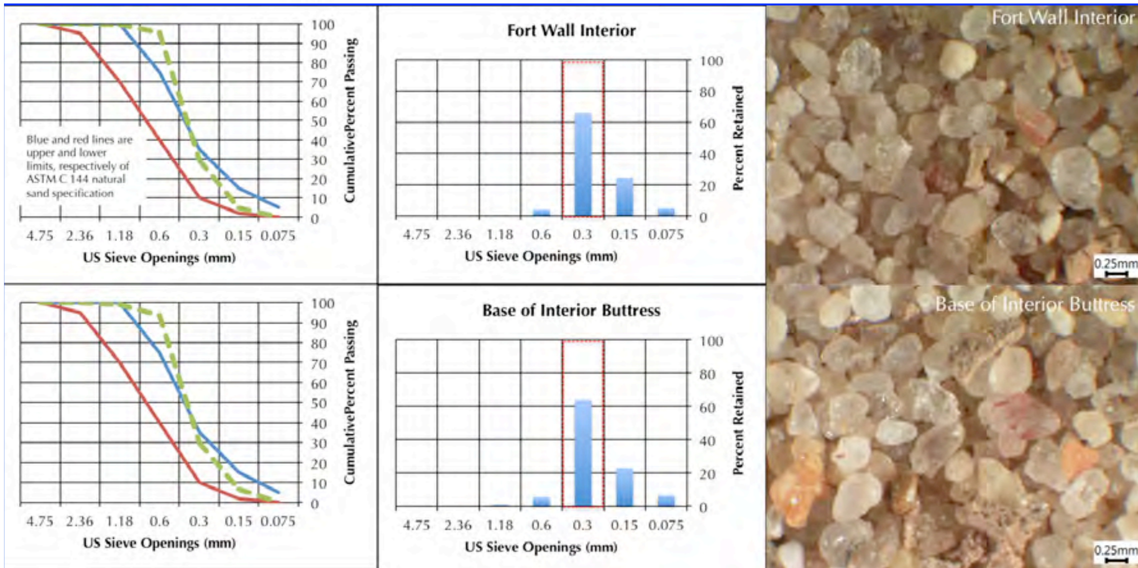


Sand gradation has a strong influence on workability, water retention, binder content, appearance, and performance of a pointing mortar.

Sand Types In Individual Sieves



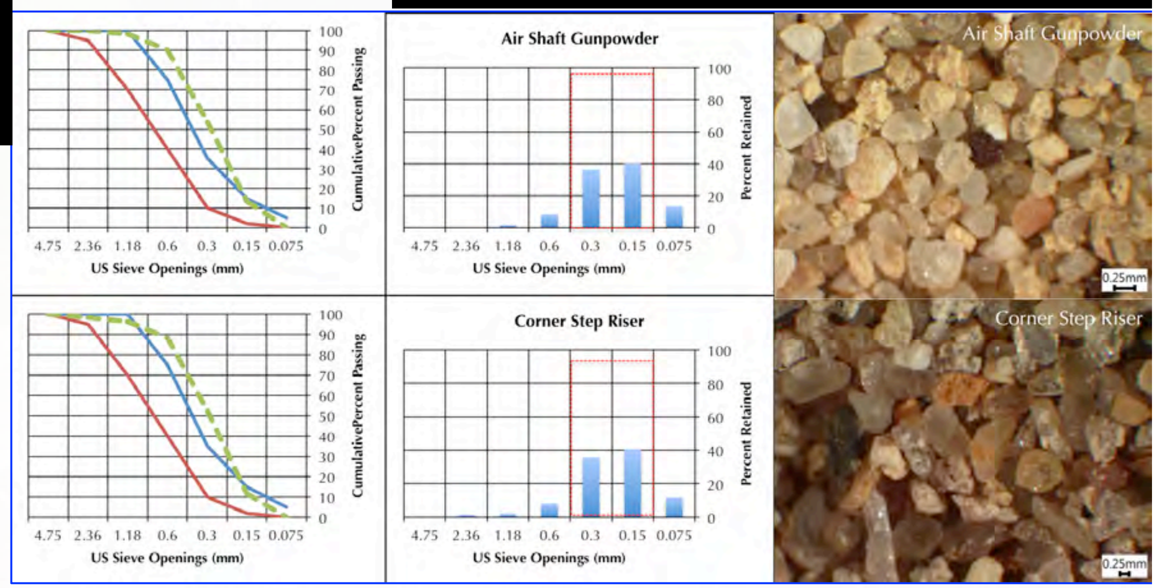
Sand Size Distribution & Color Variation



Sands used in original mortars and later pointing ones have different grain-size distribution.

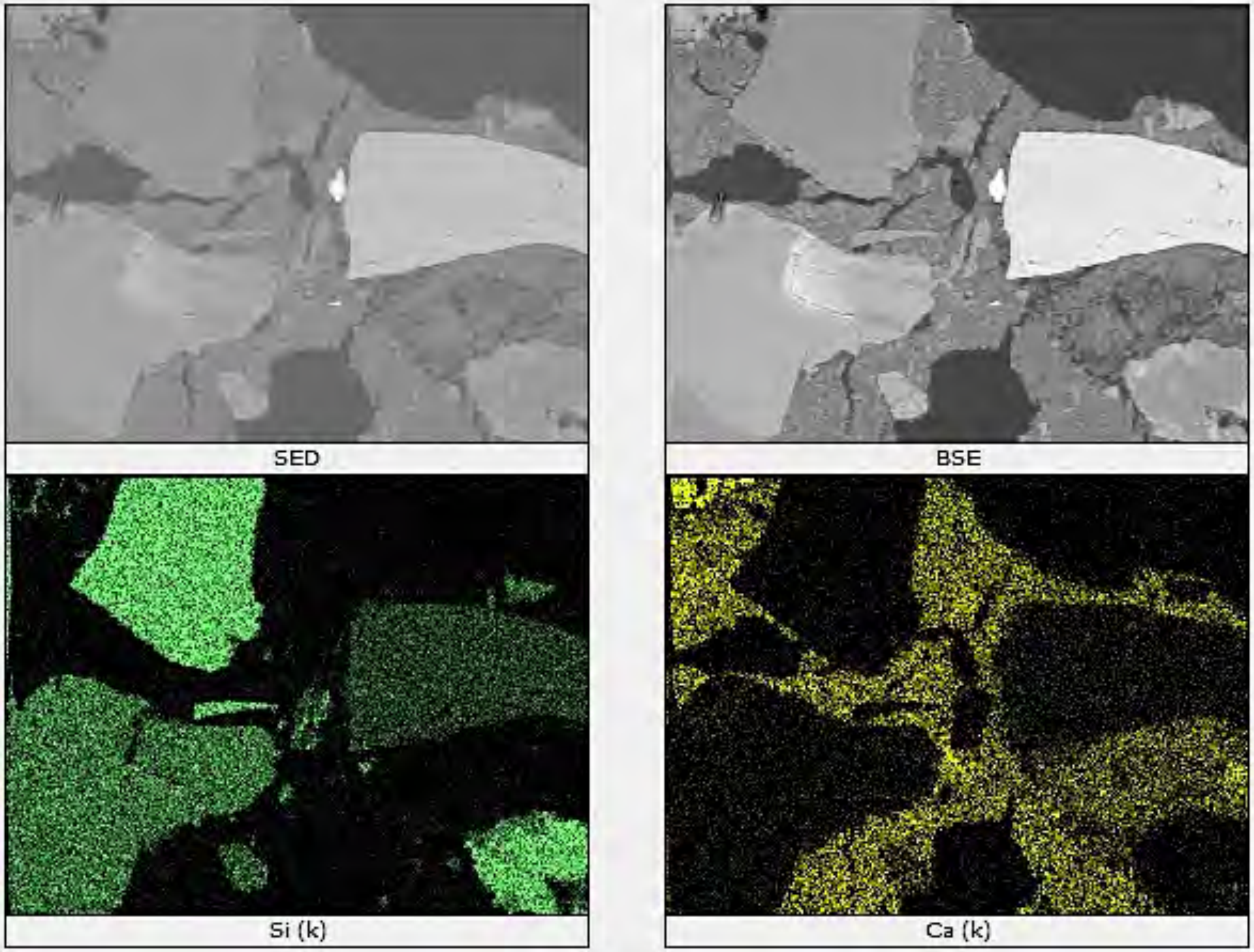
Sands used in Fort Wall Interior and Base of Buttress have very similar grain-size distribution

Sands used in Air Shafts Gunpowder and Corner Step Riser have very similar grain-size distribution



Scanning Electron Microscopy & X-Ray Microanalysis

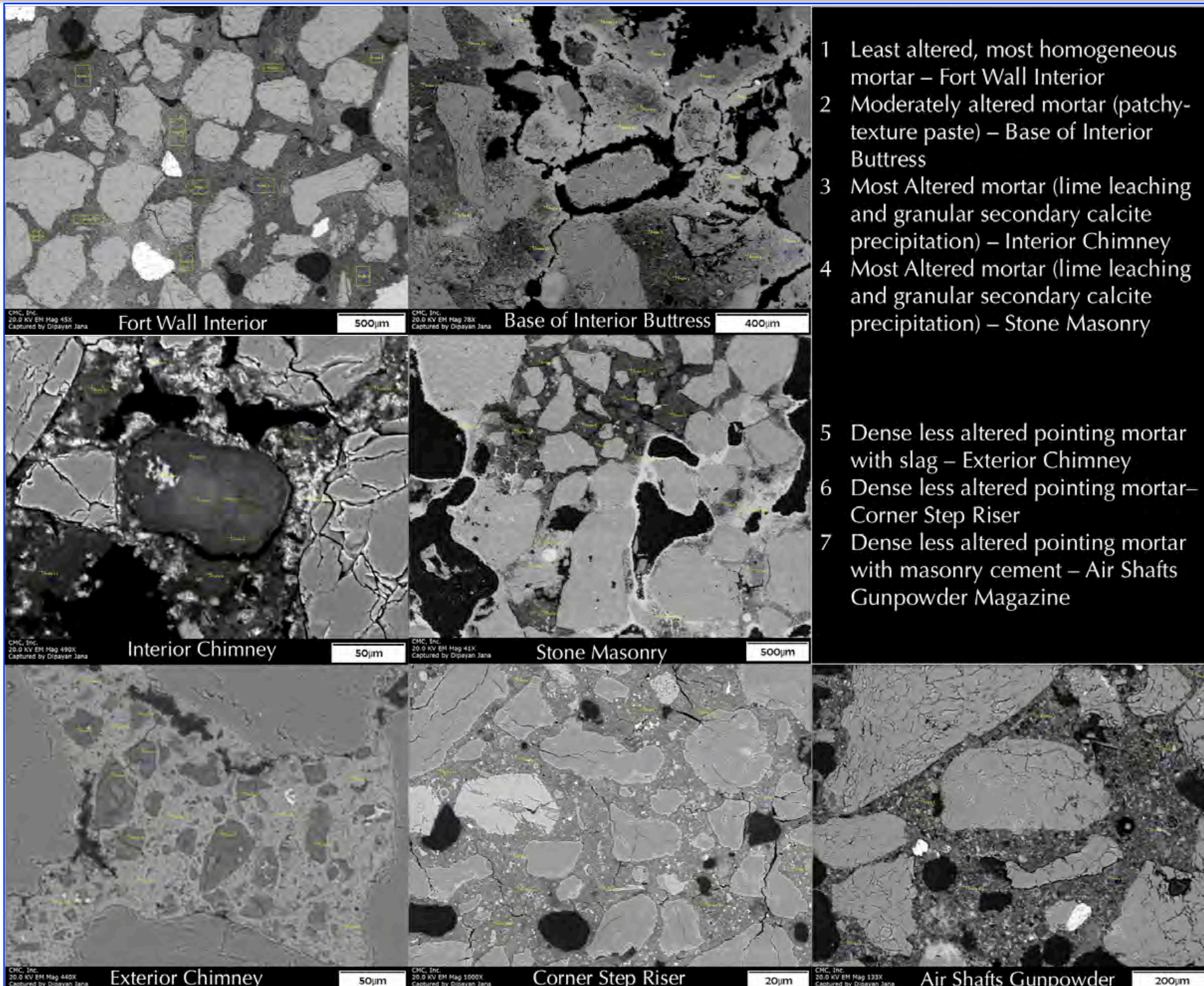
Application of SEM-EDS: Chemical Variations in Sand and Binder



Application of SEM-EDS: Microstructural Variations in Paste

Degree of alteration of paste from lime leaching to secondary calcite precipitation from darker to brighter paste patches, respectively in BSE images in Buttress, Interior Chimney, and Stone Masonry mortars.

Relatively homogeneous paste in mortars from Fort Wall, Exterior Chimney, Corner Step Riser, and Air Shafts.

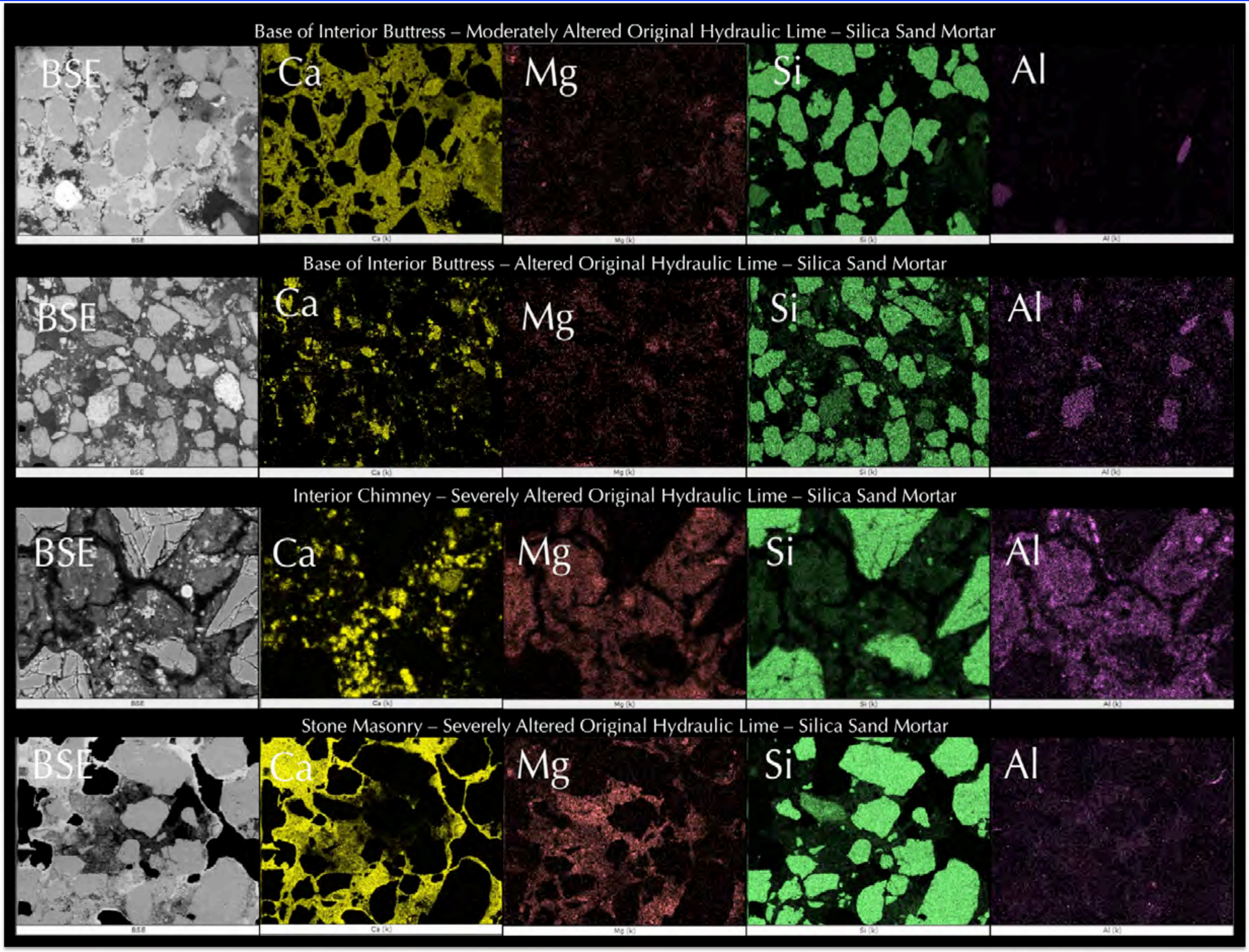


1. Least altered, most homogeneous mortar – Fort Wall Interior
2. Moderately altered mortar (patchy-texture paste) – Base of Interior Buttress
3. Most Altered mortar (lime leaching and granular secondary calcite precipitation) – Interior Chimney
4. Most Altered mortar (lime leaching and granular secondary calcite precipitation) – Stone Masonry

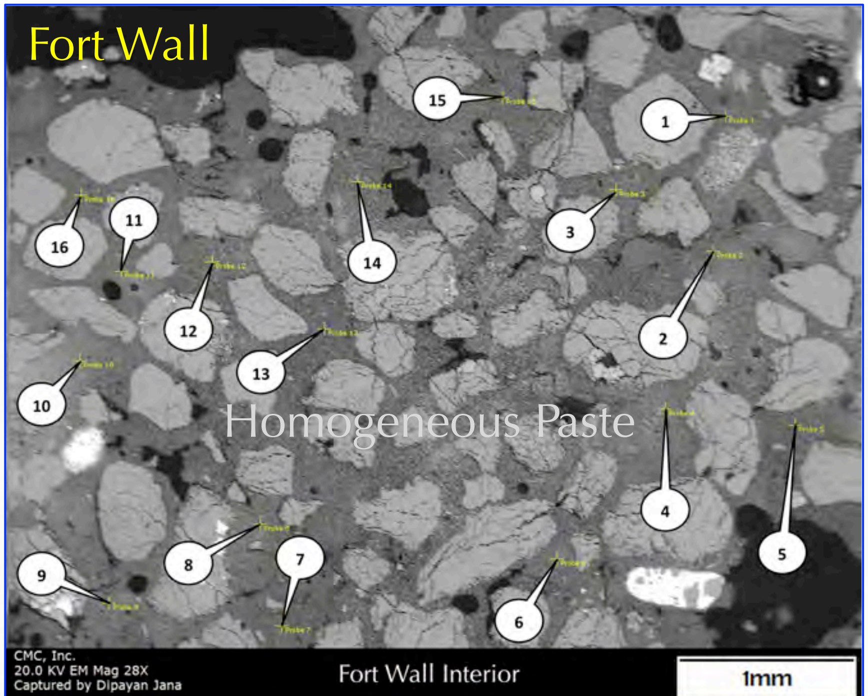
5. Dense less altered pointing mortar with slag – Exterior Chimney
6. Dense less altered pointing mortar– Corner Step Riser
7. Dense less altered pointing mortar with masonry cement – Air Shafts Gunpowder Magazine

Application of SEM-EDS: Compositional Variations in Paste From Elemental Mapping

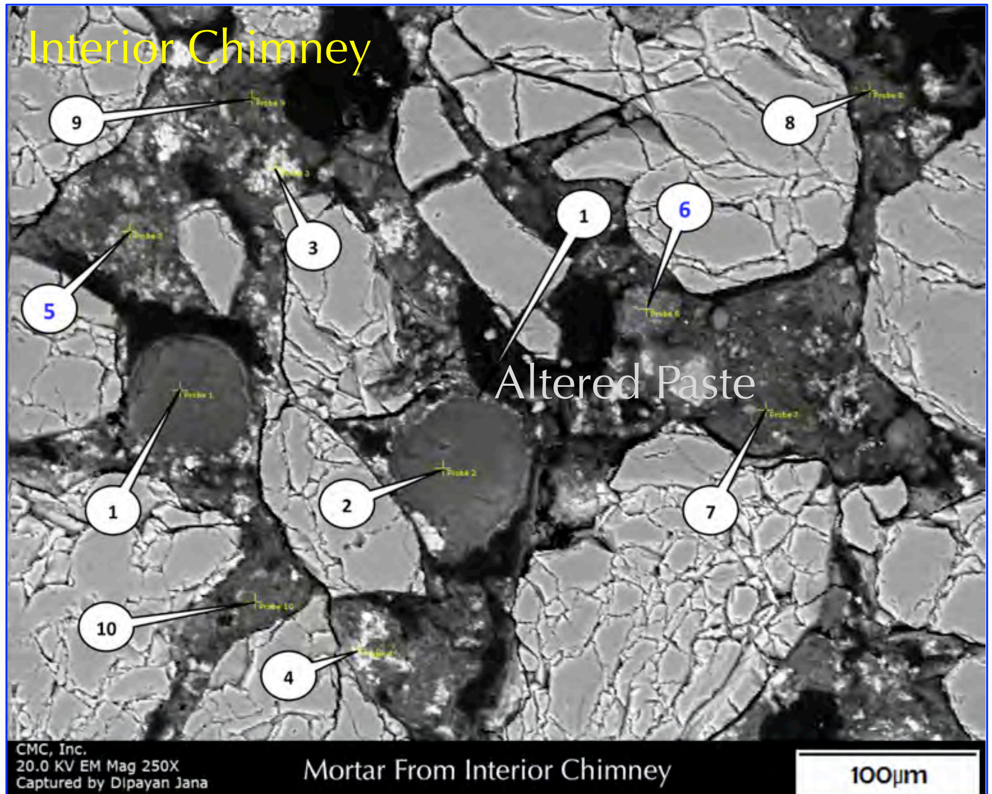
Degree of alteration of paste from lime leaching to secondary calcite precipitation from darker to brighter paste patches, respectively in dot maps of Ca in Buttress, Interior Chimney, and Stone Masonry mortars



Application of SEM-EDS: Chemical Variations in Pastes



Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	TiO2	FeO	Total	Phase	Paste CI
1	82.29	15.53	0.35	0.00	0.60	0.00	0.57	0.00	0.66	100.00	Paste	0.53
2	72.03	21.78	2.65	0.00	1.91	0.00	0.24	0.00	1.39	100.00	Paste	0.87
3	82.54	15.20	1.10	0.00	0.43	0.00	0.14	0.00	0.59	100.00	Paste	0.53
4	72.33	19.90	2.54	0.00	2.34	0.00	0.50	0.00	2.39	100.00	Paste	0.80
5	0.39	97.99	1.01	0.00	0.00	0.35	0.00	0.22	0.04	100.00	Quartz	-
6	74.11	20.51	1.99	0.00	2.47	0.22	0.35	0.00	0.34	99.99	Paste	0.77
7	0.00	97.26	1.84	0.00	0.16	0.00	0.00	0.49	0.24	99.99	Quartz	-
8	80.99	14.74	1.66	0.00	1.02	0.00	0.27	0.00	1.32	100.00	Paste	0.53
9	74.43	20.48	2.33	0.00	1.50	0.00	0.45	0.00	0.81	100.00	Paste	0.79
10	77.09	17.91	1.98	0.00	1.78	0.00	0.46	0.00	0.78	100.00	Paste	0.66
11	72.59	19.96	2.20	0.00	3.00	0.00	0.68	0.00	1.57	100.00	Paste	0.77
12	83.37	13.36	0.68	0.00	1.74	0.00	0.00	0.44	0.40	99.99	Paste	0.45
13	82.03	15.60	1.36	0.00	0.14	0.00	0.39	0.00	0.48	100.00	Paste	0.55
14	75.43	18.82	3.11	0.00	1.43	0.00	0.31	0.00	0.89	99.99	Paste	0.73
15	73.06	19.91	2.61	0.00	2.25	0.00	0.28	0.00	1.88	99.99	Paste	0.79
16	76.85	18.44	2.16	0.00	0.69	0.00	0.39	0.02	1.44	99.99	Paste	0.71



Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	P2O5	TiO2	FeO	Total	Phase	Paste CI
1	0.17	42.59	18.51	0.47	19.28	3.09	0.02	14.69	0.00	1.17	99.99	Si-Al-Mg-P grain	5.17
2	0.14	41.41	17.41	0.00	13.82	7.23	0.00	19.54	0.00	0.44	99.99		6.95
3	85.74	4.96	2.55	0.00	1.06	1.24	0.66	1.78	0.45	1.57	100.01	Secondary calcite	0.20
4	74.94	7.09	5.76	0.00	4.15	1.66	0.31	2.35	0.42	3.32	100.00		0.35
5	44.37	26.77	7.45	0.00	11.52	3.00	0.00	3.40	0.40	3.09	100.00	Ca-Si-Mg Paste	1.41
6	44.21	27.80	7.52	0.00	12.19	2.79	0.07	3.04	0.00	2.39	100.01		1.43
7	2.51	41.65	16.27	0.00	13.34	6.87	0.00	15.02	0.05	4.29	100.00	Altered (Si-Al-Mg-P) Paste	6.49
8	0.49	48.52	15.83	0.00	13.33	5.16	0.34	10.43	0.11	5.79	100.00		8.21
9	5.09	47.75	15.14	0.00	6.88	6.99	0.67	11.33	0.12	6.03	100.00		10.5
10	0.48	44.44	18.70	0.00	12.09	5.37	0.11	14.69	0.04	4.09	100.01		8.50

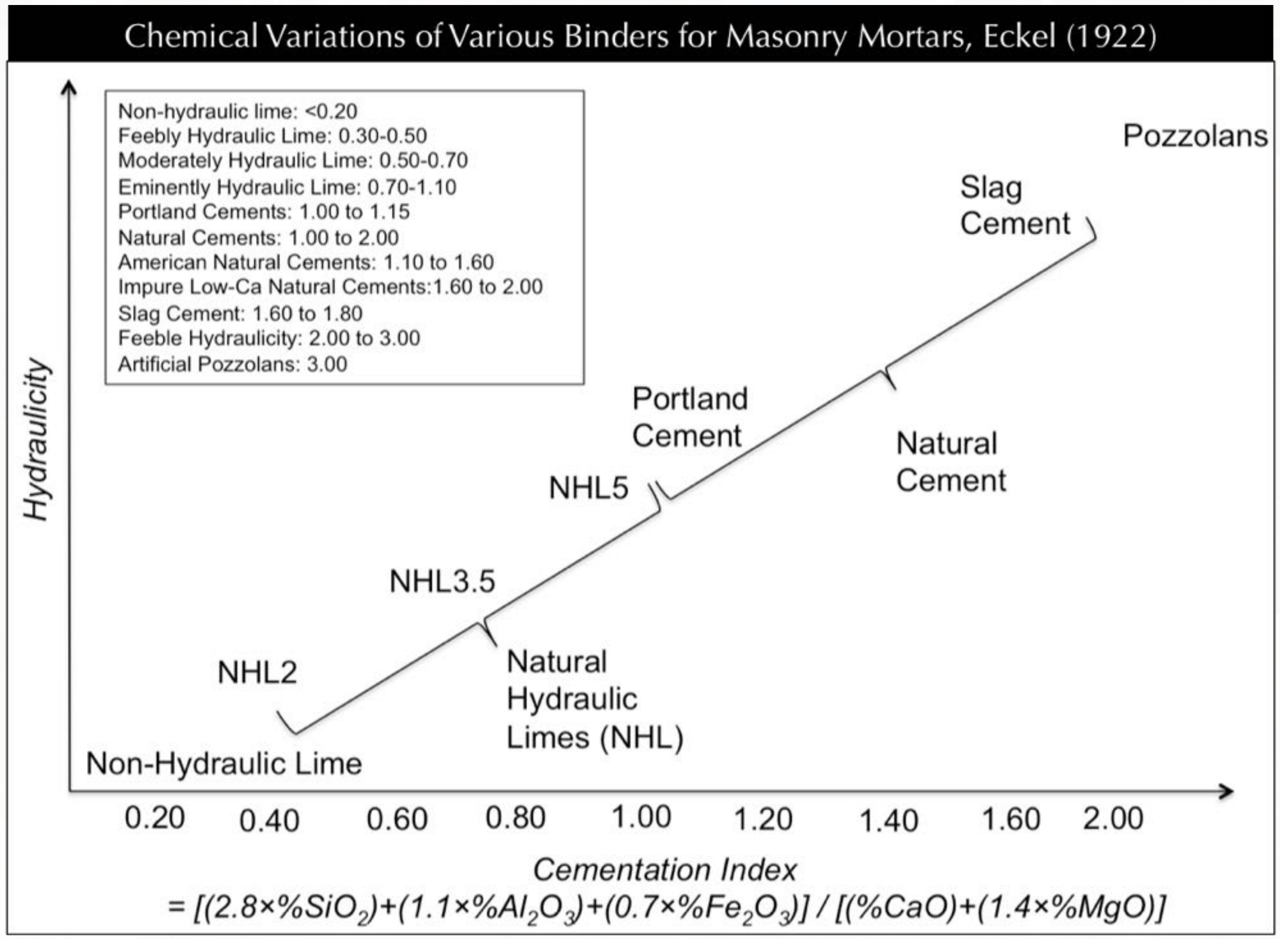
Application of SEM-EDS: Chemical Variations in Paste from Cementation Index, CI (Eckel 1922)

Cementation Index (CI, Eckel 1922) =

$[(2.8 \times SiO_2) + (1.1 \times Al_2O_3) + (0.7 \times Fe_2O_3)]$

divided by

$[(CaO) + (1.4 \times MgO)]$

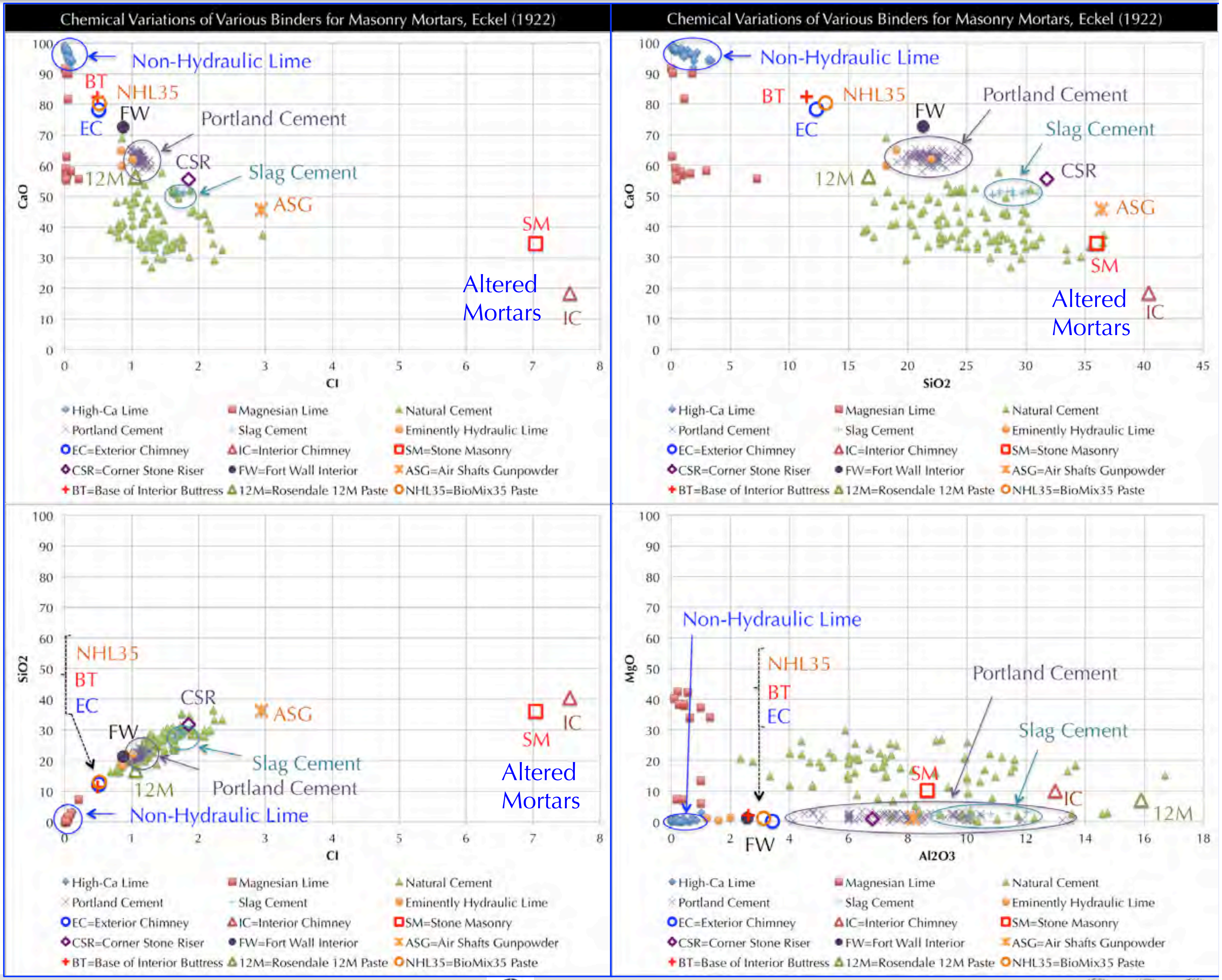


Application of SEM-EDS:

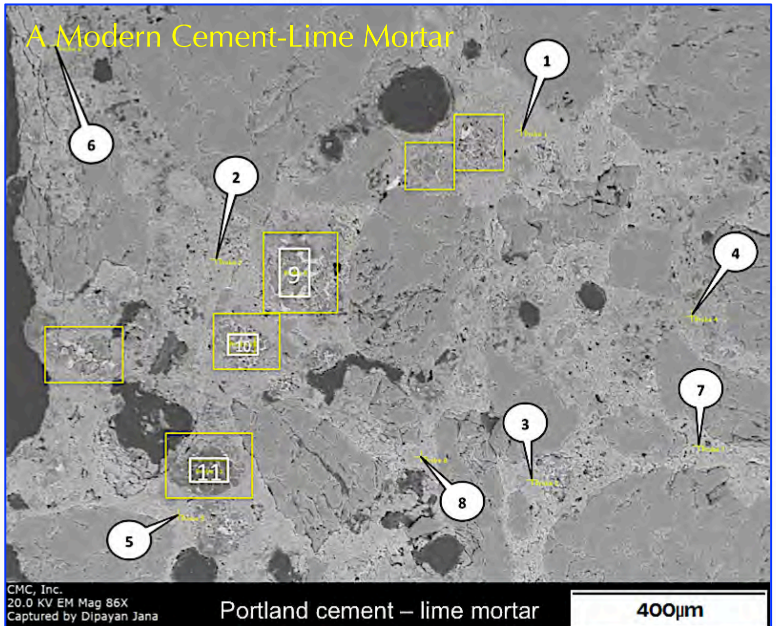
Chemical Variations In Paste Of The Present Mortars

Compared To -

Common Masonry Binders From Eckel (1922)



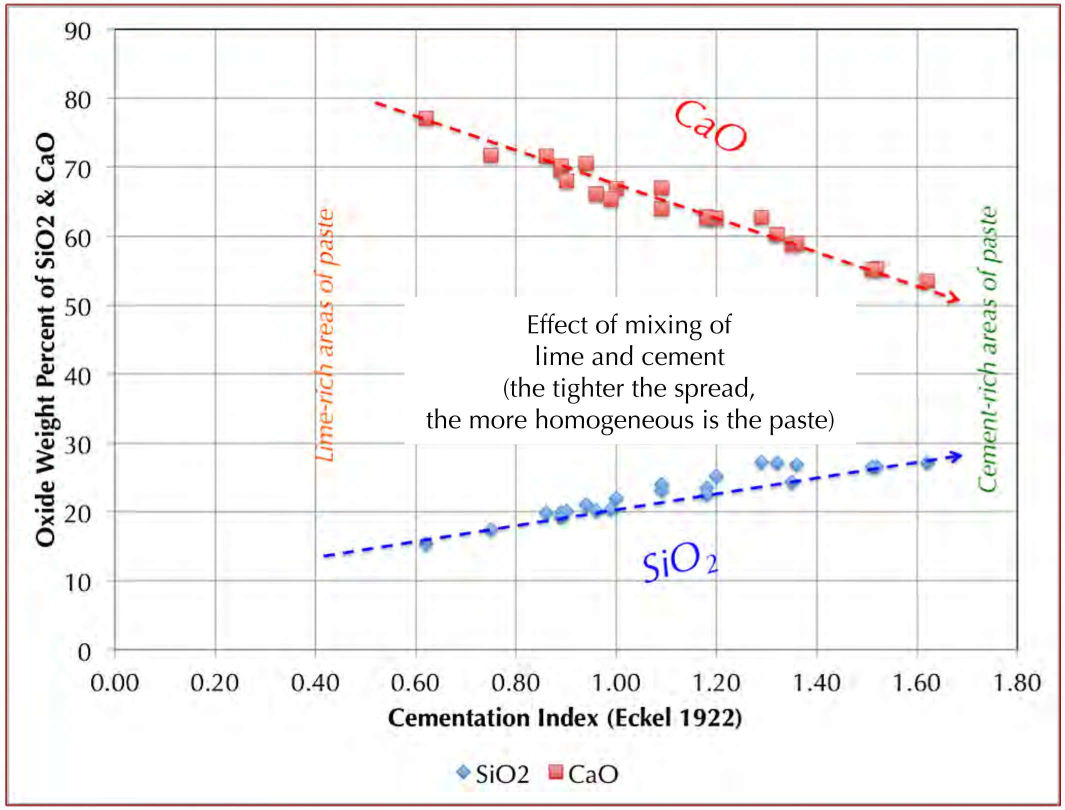
Application of SEM-EDS: Chemical Variations in Portland Cement-Lime Mortar Paste



Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	TiO2	FeO	Total	Phase	Paste CI
1	80.96	12.23	3.60	0.00	0.16	0.00	1.11	0.90	1.03	99.99	Paste	0.48
2	90.83	6.47	1.77	0.00	0.12	0.00	0.33	0.00	0.47	99.99	Paste	0.22
3	54.57	18.91	6.43	0.00	1.61	0.00	1.31	1.16	16.01	100.00	RCP	-
4	12.28	49.26	5.14	0.23	2.72	0.00	7.36	0.00	23.01	100.00	RCP	-
5	80.25	11.99	4.00	0.00	0.30	0.00	1.89	0.04	1.53	100.00	Paste	0.48
6	86.83	9.09	1.98	0.00	0.07	0.00	0.86	0.21	0.96	100.00	Paste	0.33
7	87.47	8.23	2.02	0.00	0.21	0.00	0.54	0.00	1.52	99.99	Paste	0.30
8	88.78	6.86	2.34	0.00	0.48	0.00	0.71	0.00	0.83	100.00	Paste	0.25
9	36.36	38.04	12.39	0.33	1.54	0.00	1.34	1.24	8.75	99.99	RCP	-
10	32.64	43.86	10.19	0.59	1.27	0.00	1.57	1.07	8.81	100.00	RCP	-
11	15.41	60.51	13.76	0.14	1.13	0.00	1.60	1.41	6.03	99.99	RCP	-

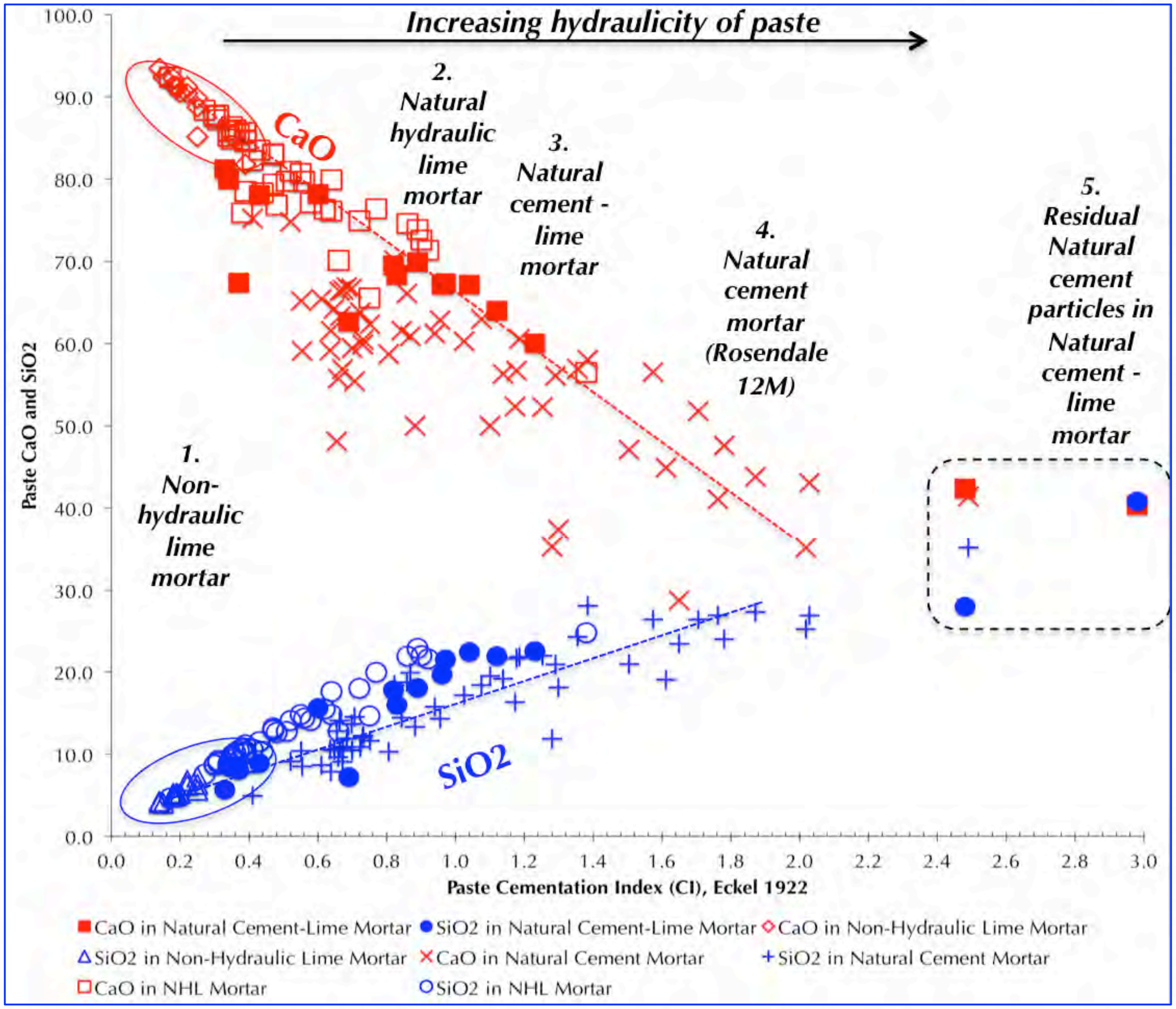
SEM-EDS analysis of a paste fraction of a cement-lime mortar. Yellow boxes show residual cement particles, white boxes show area-mode analysis of residual cement particles and tips of callouts show locations of analysis of paste. The Table below shows results of analysis of paste and residual cement particles (RCPs).

CI, or Cementation Index, after Eckel 1922, which is $[(2.8*SiO_2+1.1*Al_2O_3+0.7*Fe_2O_3)/(CaO+1.4*MgO)]$ is a parameter of hydraulicity of a mortar, e.g., a lime mortar has a CI of < 1 whereas a cement-lime mortar has a CI of >1. Hence determination of CI of a mortar's paste from SEM-EDS analysis can provide the first-hand clue of its hydraulicity. CI of paste progressively increases from non-hydraulic lime mortar to hydraulic lime mortar, natural cement-lime mortar, natural cement mortar, and Portland cement – lime mortar.

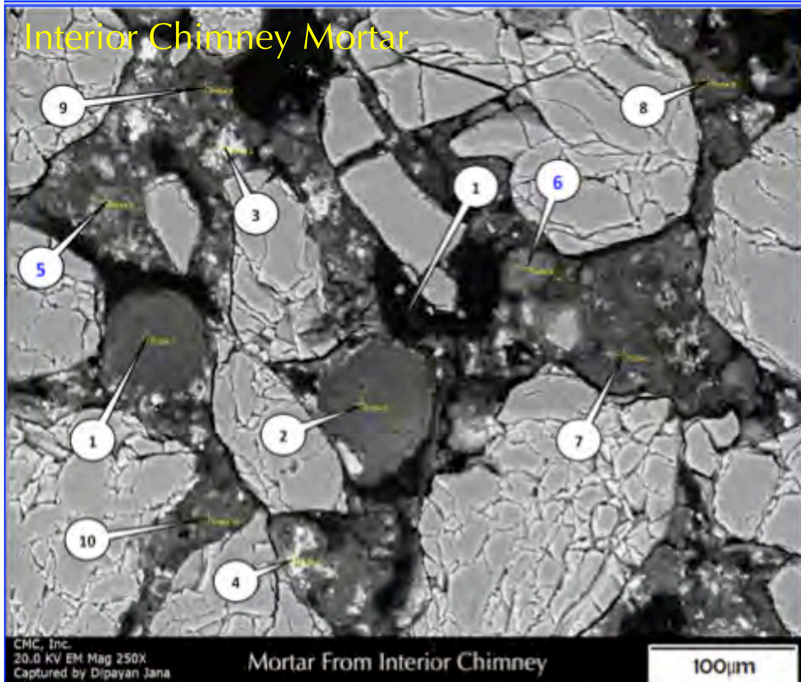


Application of SEM-EDS: Chemical Variations of Pastes in:

1. A Historic Non-Hydraulic Lime Mortar;
2. A Modern Natural Hydraulic Lime Mortar (BioMix 35 having NHL 3.5);
3. A Historic Natural Cement – Lime Mortar
4. A Modern Natural Cement Mortar (Rosendale 12M)
5. Residual Natural Cement Particles

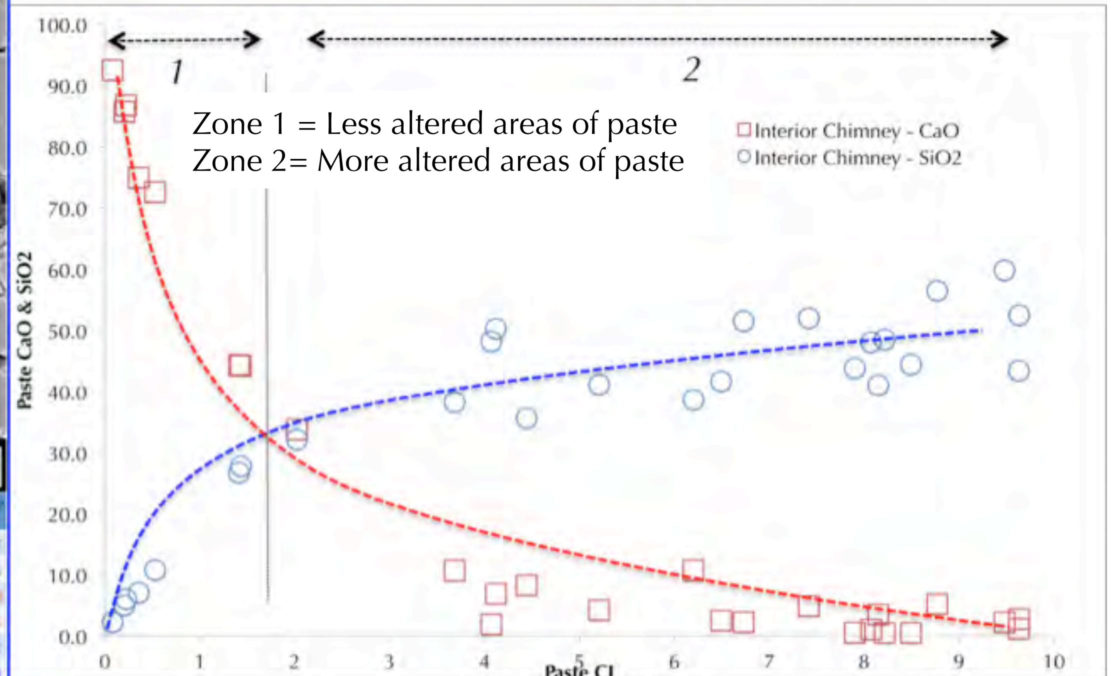


Application of SEM-EDS: Effects of Alterations In Chemical Composition of Paste



Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	P2O5	TiO2	FeO	Total	Phase	Paste CI
1	0.17	42.59	18.51	0.47	19.28	3.09	0.02	14.69	0.00	1.17	99.99	Si-Al-Mg-P grain	5.17
2	0.14	41.41	17.41	0.00	13.82	7.23	0.00	19.54	0.00	0.44	99.99		6.95
3	85.74	8.96	2.51	0.00	3.06	1.24	0.06	1.70	0.05	3.57	100.01	Secondary calcite	0.20
4	74.94	7.09	5.76	0.00	4.13	1.66	0.41	2.35	0.42	3.32	100.00		0.15
5	44.37	26.77	7.45	0.00	11.52	3.00	0.00	3.40	0.40	3.09	100.00	Ca-Si-Mg Paste	1.41
6	44.21	27.80	7.52	0.00	12.19	2.79	0.07	3.04	0.00	2.39	100.01		1.43
7	2.51	41.65	16.27	0.00	13.34	6.87	0.00	15.02	0.05	4.29	100.00	Altered (Si-Al-Mg-P) Paste	6.49
8	0.49	48.52	15.83	0.00	13.33	5.16	0.34	10.43	0.11	5.79	100.00		8.21
9	5.09	47.75	15.14	0.00	6.88	6.99	0.67	13.33	0.12	6.03	100.00		10.5
10	0.48	44.44	18.70	0.00	12.09	5.37	0.11	14.69	0.04	4.09	100.01		8.50

Unlike an unaltered modern cement-lime mortar, an altered historic mortar shows significant spread in paste-CI due to various alterations during service, e.g., lime leaching (causing very high CI) to secondary calcite precipitation (causing very low CI).

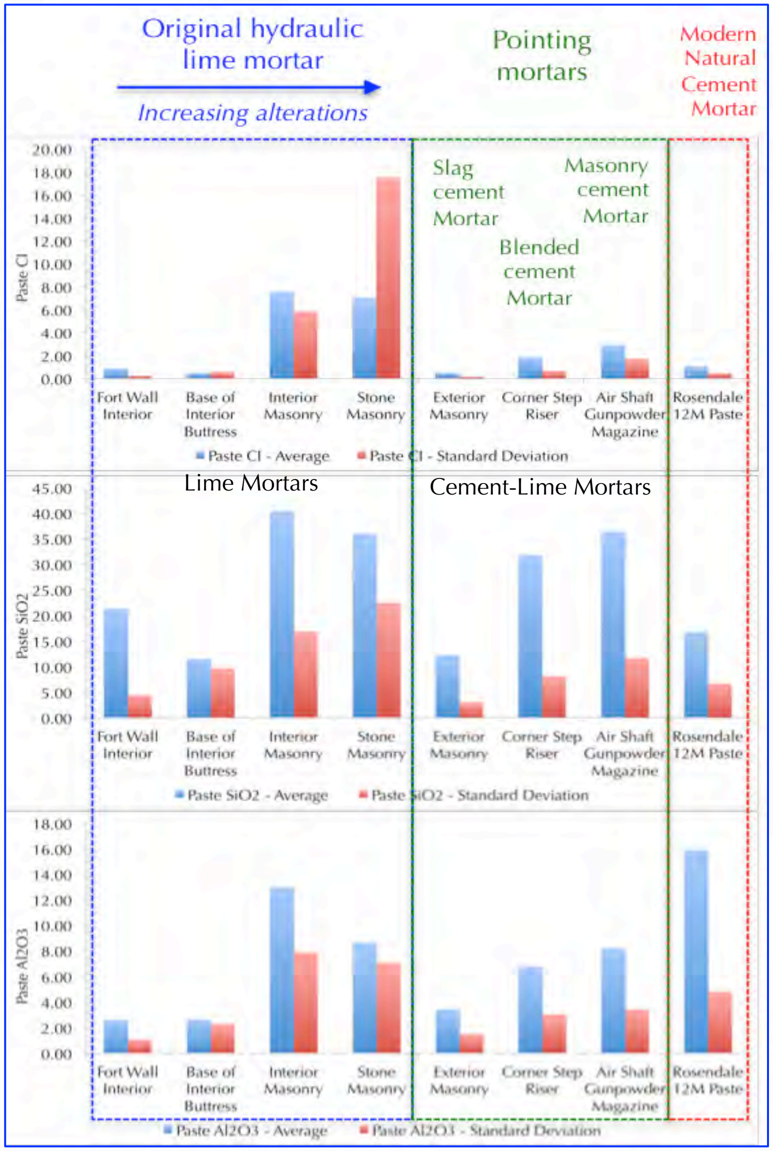


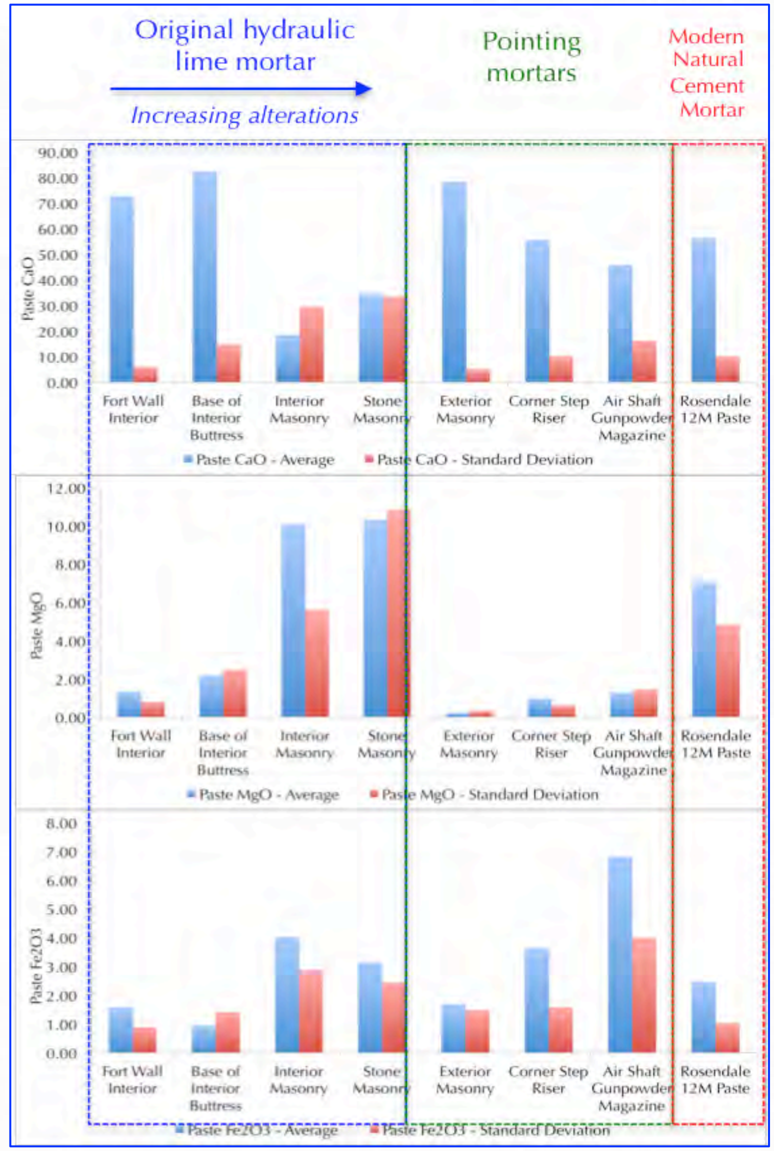
Region 1: Effect of mixing between lime (Ca)-rich and cement (Si, CI)-rich components;
Region 2: Effect of secondary alterations, e.g., lime leaching, secondary calcite precipitation, etc. that causes enrichment of silica, magnesia, alumina at the expense of calcium

Application of SEM-EDS: Chemical Variations in Altered Paste

Measuring degree of alterations of paste from standard deviations of paste compositions from SEM-EDS.

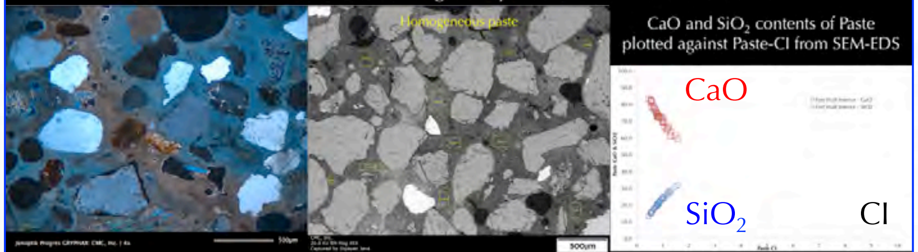
The higher the red bars, the more altered are the pastes.





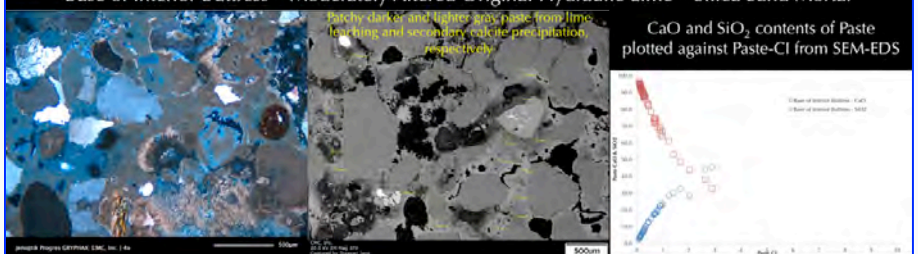
Application of SEM-EDS: Microstructural & Chemical Variations in Pastes

Fort Wall Interior – Least Altered Original Hydraulic Lime – Silica Sand Mortar



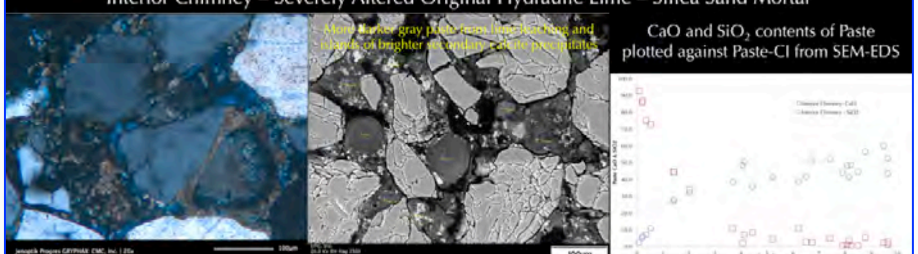
CaO and SiO_2 contents of Paste plotted against Paste-CI from SEM-EDS

Base of Interior Buttress – Moderately Altered Original Hydraulic Lime – Silica Sand Mortar



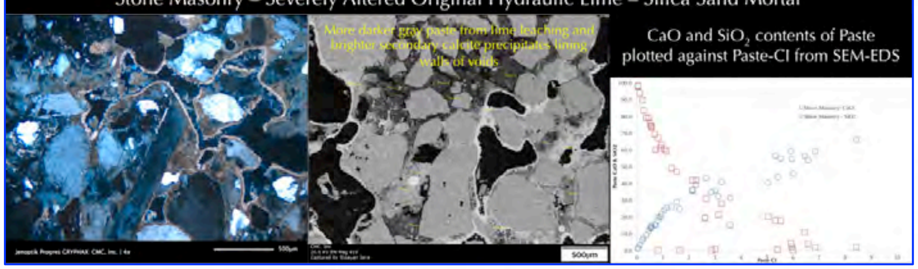
CaO and SiO_2 contents of Paste plotted against Paste-CI from SEM-EDS

Interior Chimney – Severely Altered Original Hydraulic Lime – Silica Sand Mortar



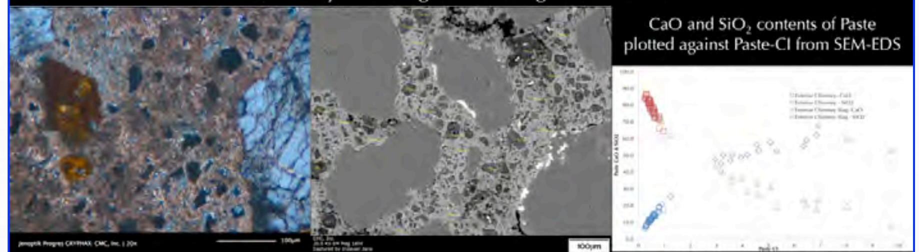
CaO and SiO_2 contents of Paste plotted against Paste-CI from SEM-EDS

Stone Masonry – Severely Altered Original Hydraulic Lime – Silica Sand Mortar



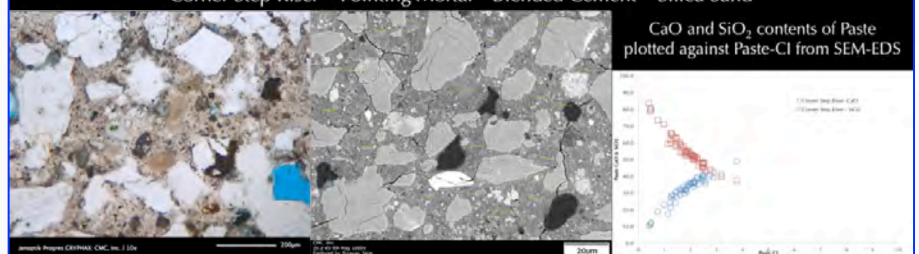
CaO and SiO_2 contents of Paste plotted against Paste-CI from SEM-EDS

Exterior Chimney – Pointing Mortar - Slag Cement – Silica Sand



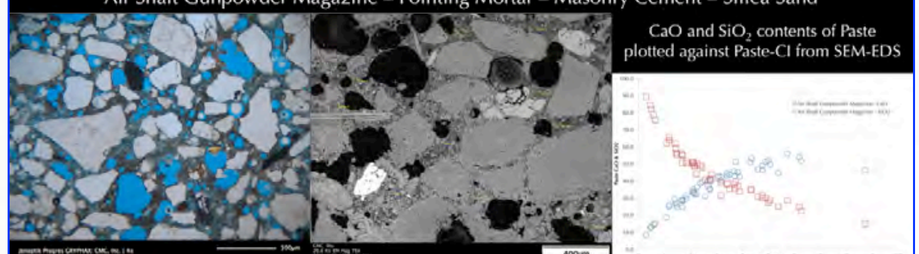
CaO and SiO_2 contents of Paste plotted against Paste-CI from SEM-EDS

Corner Step Riser – Pointing Mortar - Blended Cement – Silica Sand



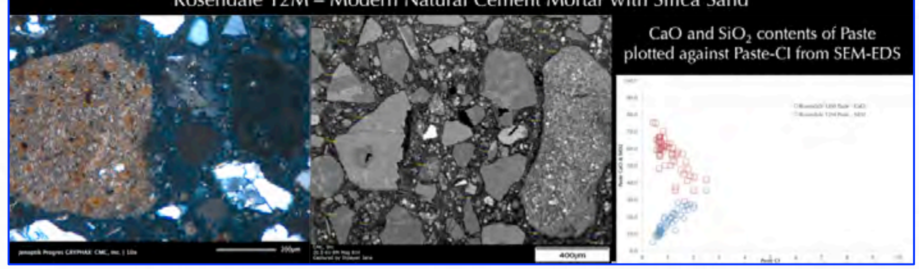
CaO and SiO_2 contents of Paste plotted against Paste-CI from SEM-EDS

Air Shaft Gunpowder Magazine – Pointing Mortar – Masonry Cement – Silica Sand



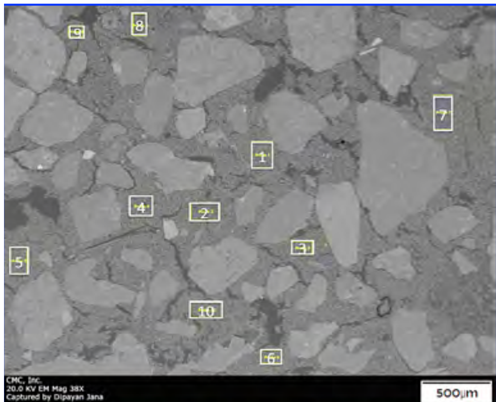
CaO and SiO_2 contents of Paste plotted against Paste-CI from SEM-EDS

Rosendale 12M – Modern Natural Cement Mortar with Silica Sand



CaO and SiO_2 contents of Paste plotted against Paste-CI from SEM-EDS

Applications of SEM-EDS: Chemical Variations in Pastes of High-Ca Lime Mortar vs. Dolomitic Lime Mortar vs. Hydraulic Lime Mortar vs. Natural Cement – Lime Mortar



Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	TiO2	FeO	Total	Phase	Paste CI
1	93.70	4.20	0.35	0.00	0.67	0.00	0.03	0.12	0.92	99.99	Paste	0.14
2	92.52	3.83	1.11	0.00	1.26	0.00	0.12	0.29	0.88	100.01	Paste	0.13
3	92.75	4.21	0.96	0.00	1.45	0.00	0.25	0.00	0.38	100.00	Paste	0.14
4	92.76	4.55	1.13	0.00	1.27	0.00	0.07	0.00	0.22	100.00	Paste	0.15
5	93.38	3.68	0.74	0.00	1.48	0.00	0.15	0.00	0.57	100.00	Paste	0.12
6	94.30	2.86	0.73	0.00	0.56	0.00	0.27	0.17	1.11	100.00	Paste	0.10
7	84.08	9.86	1.65	0.00	2.59	0.00	0.11	0.00	1.70	99.99	Paste	0.35
8	94.04	4.29	0.79	0.00	0.60	0.00	0.03	0.00	0.25	100.00	Paste	0.14
9	91.92	4.07	1.23	0.00	1.07	0.00	0.04	0.07	1.59	99.99	Paste	0.15
10	92.51	3.33	1.39	0.00	0.81	0.00	0.13	0.00	1.82	99.99	Paste	0.13

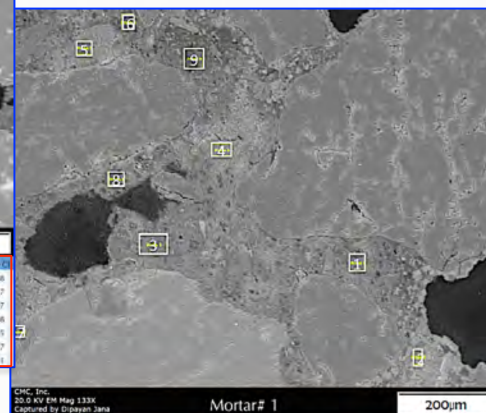
1. Non-Hydraulic High-Ca Lime Mortar

- Lowest CI
- Highest CaO
- Lowest SiO_2, MgO

Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	TiO2	FeO	Total	Phase	Paste CI
1	78.03	2.58	0.71	0.62	17.70	0.01	0.35	0.00	0.65	99.99	Paste	0.08
2	78.54	2.05	0.79	0.41	18.18	0.02	0.02	0.07	0.34	100.00	Paste	0.07
3	81.18	2.54	0.03	0.81	14.70	0.02	0.72	0.00	0.04	99.99	Paste	0.07
4	84.03	1.97	1.41	0.50	11.97	0.01	0.11	1.66	1.66	99.99	Paste	0.08
5	80.97	1.37	0.85	0.85	15.78	0.01	0.17	0.00	1.10	99.99	Paste	0.05
6	78.72	2.11	0.99	1.06	17.07	0.01	0.03	0.37	0.68	100.00	Paste	0.07
7	88.19	0.02	0.44	1.37	9.55	0.01	0.41	1.03	0.56	99.99	Paste	0.01

2. Non-Hydraulic Dolomitic Lime Mortar

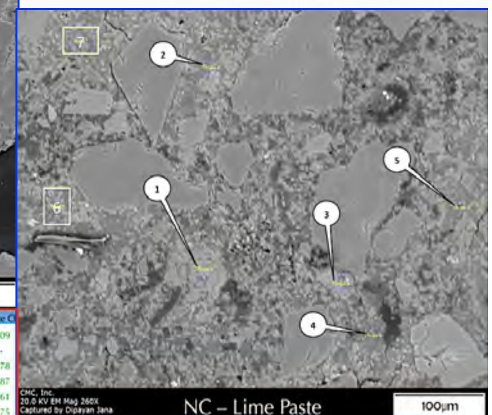
- Lowest CI
- Characteristically high MgO
- Lowest SiO_2



Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	TiO2	FeO	Total	Phase	Paste CI
1	50.82	36.79	7.05	0.00	1.61	0.00	0.55	3.05	0.12	99.99	Paste	2.09
2	57.56	7.65	3.97	0.00	20.28	0.06	1.64	8.26	0.57	99.99	Mg-rich	-
3	51.50	32.50	9.99	0.00	4.33	0.00	0.40	0.62	0.65	99.99	Paste	1.78
4	59.28	19.45	2.88	0.70	6.06	0.00	0.27	9.89	1.47	100.00	Paste	0.87
5	44.11	36.01	14.47	0.00	0.53	0.00	3.04	1.11	0.73	100.00	Paste	2.61
6	46.95	28.42	12.45	0.11	4.96	3.41	0.62	1.21	1.85	100.00	Paste	1.75
7	36.22	43.77	11.65	1.21	0.21	0.00	0.47	4.57	1.88	100.00	Paste	3.74
8	50.37	36.81	8.68	0.00	1.68	0.00	0.75	0.67	1.03	99.99	Paste	2.15
9	51.46	35.37	7.68	0.00	1.37	0.00	0.11	0.17	1.85	100.01	Paste	1.96

3. Hydraulic Lime Mortar

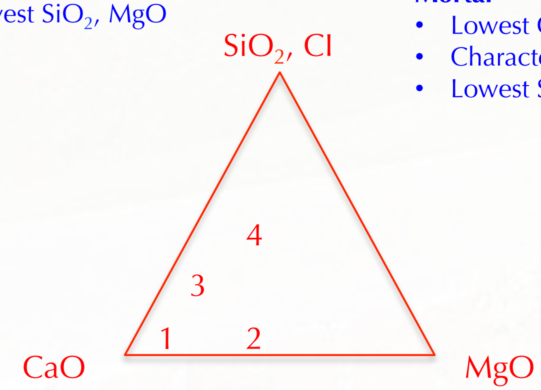
- Characteristically high SiO_2
- CI >1



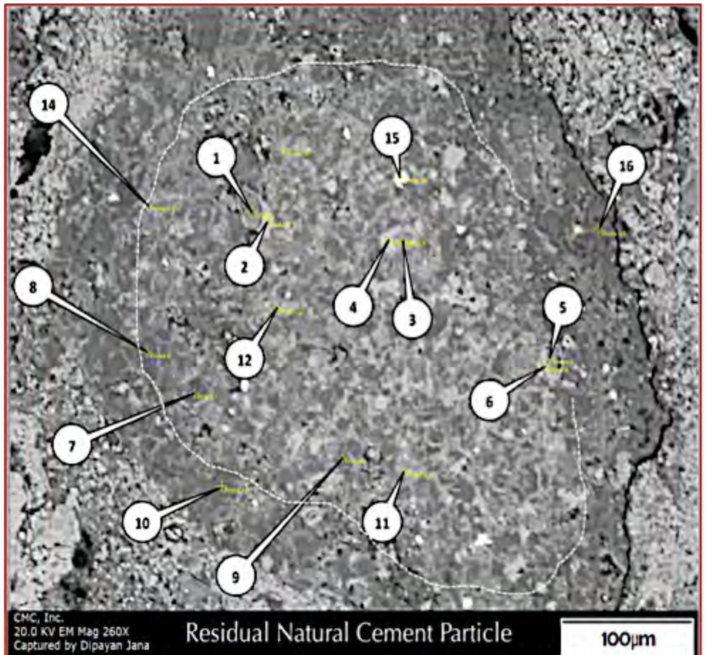
Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	TiO2	FeO	Total	Phase	Paste CI
1	77.53	8.36	1.22	0.00	7.56	0.00	0.07	0.00	5.26	100.00	Ca-rich Mg-Al-Fe-Silicate	0.12
2	20.91	50.65	8.43	0.14	13.88	0.00	0.65	0.21	5.12	100.01	Ca-poor Mg-Al-Fe-Silicate	1.81
3	75.00	6.47	2.65	0.00	8.78	0.00	0.61	0.36	6.12	99.99	Ca-rich Mg-Al-Fe-Silicate	0.29
4	16.14	49.56	11.61	0.13	14.05	0.00	0.29	0.58	7.64	100.00	Ca-poor Mg-Al-Fe-Silicate	4.18
5	13.12	57.06	9.16	0.21	11.54	0.00	0.55	1.19	7.16	99.99	Ca-poor Mg-Al-Fe-Silicate	5.97
6	37.64	37.71	6.92	0.07	11.82	0.00	0.68	0.77	4.40	100.01	Ca-Mg-Al-Silicate	2.15
7	41.27	35.72	6.96	0.18	10.57	0.00	1.13	0.00	4.16	99.99	Ca-Mg-Al-Silicate	1.97

4. Natural Cement - Lime Mortar

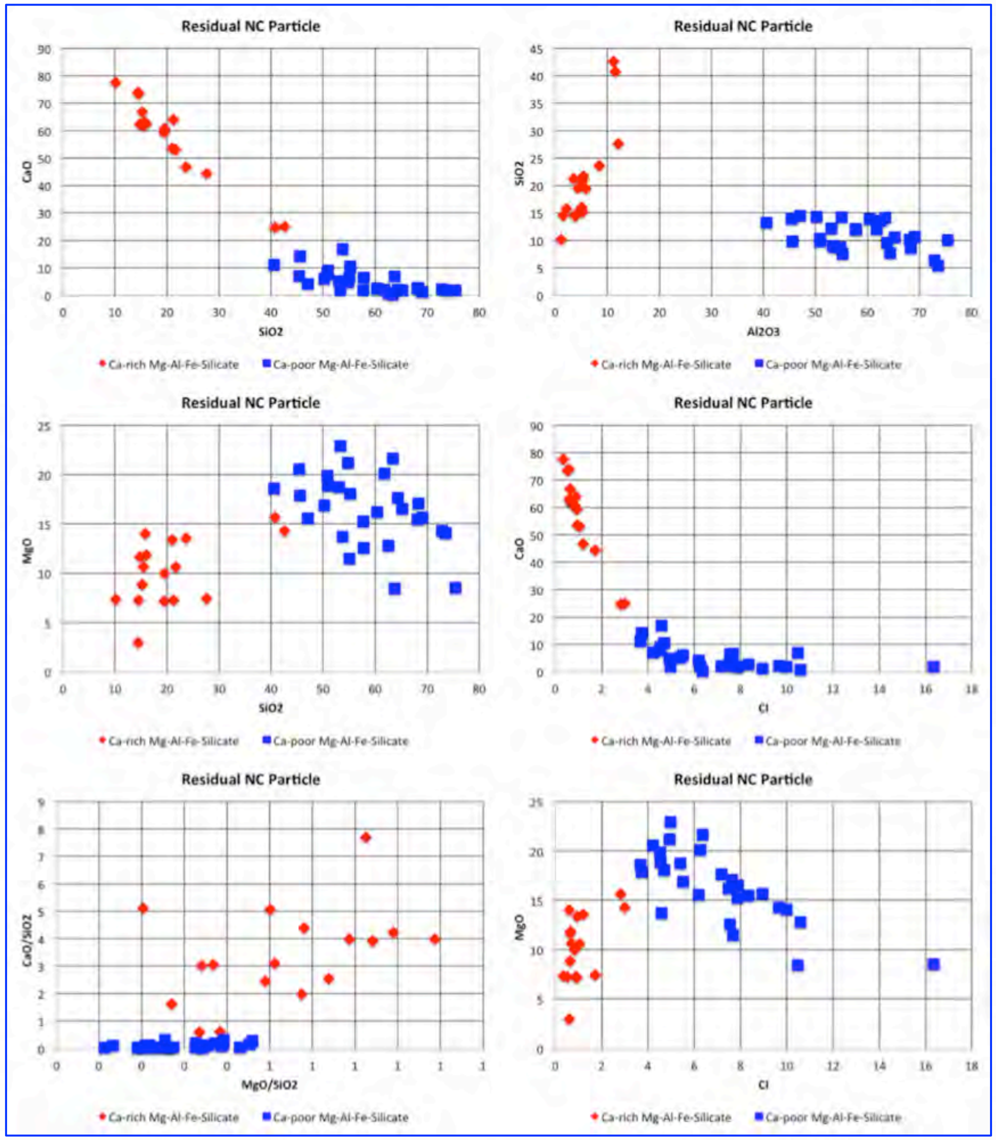
- High MgO and SiO_2
- CI <1 for Ca-rich areas and >1 for Ca-poor areas



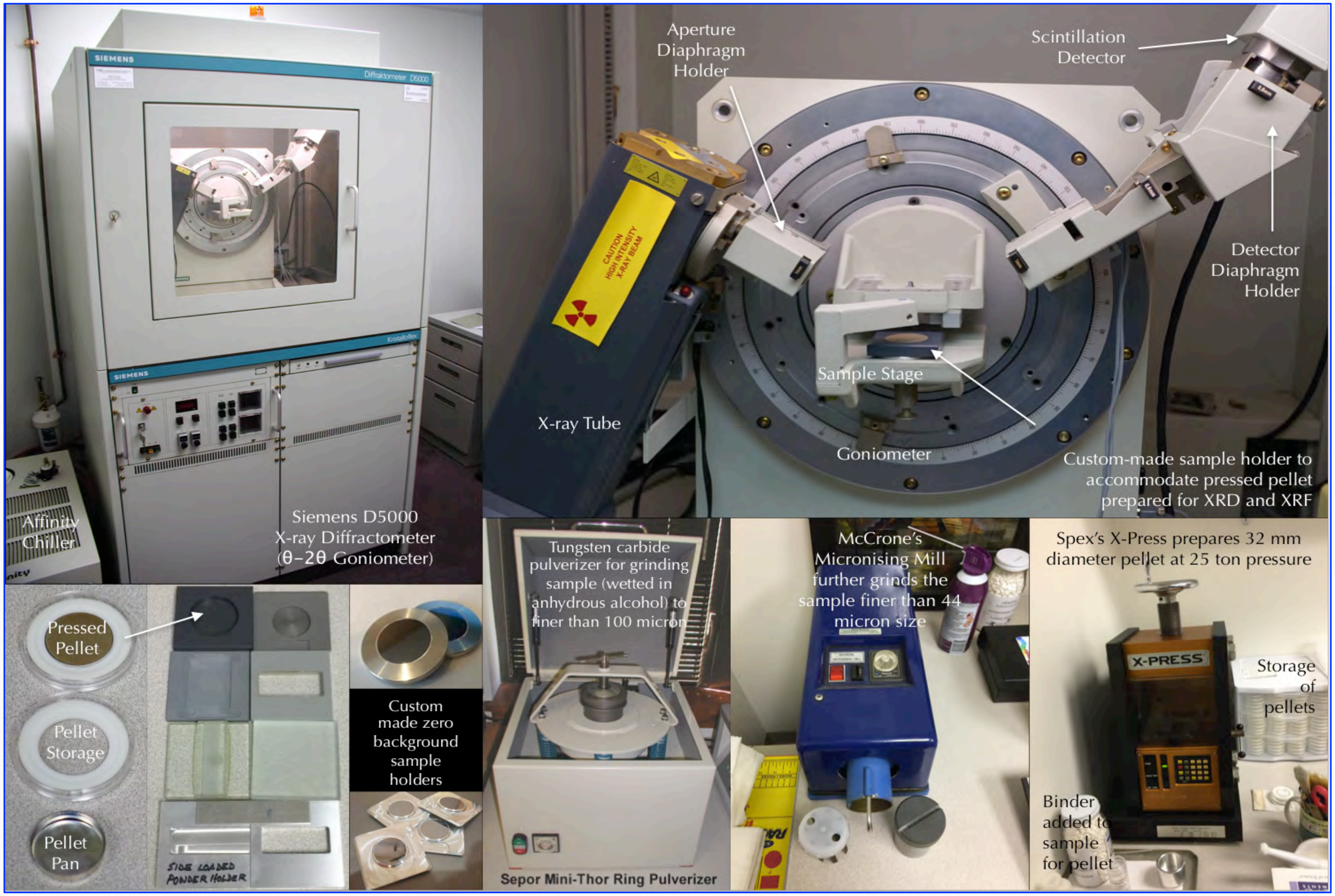
Application of SEM-EDS: Chemical Variation in Residual Natural Cement



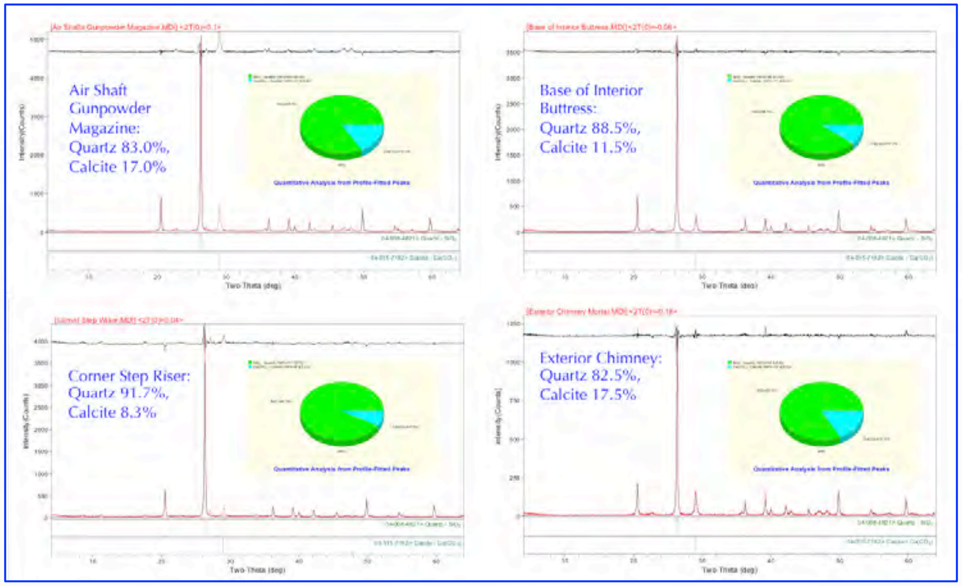
Probe	CaO	SiO2	Al2O3	Na2O	MgO	SO3	K2O	TiO2	FeO	Total	Phase	Paste CI
1	0.57	96.95	1.11	0.00	0.12	1.24	0.00	0.00	0.00	99.99	Si-Core	-
2	93.79	4.75	0.39	0.00	0.24	0.00	0.40	0.00	0.42	99.99	Ca-rim	-
3	1.40	95.51	1.40	0.00	0.04	0.35	0.00	0.66	0.63	99.99	Si-Core	-
4	94.31	4.01	0.58	0.00	0.21	0.00	0.53	0.36	0.00	100.00	Ca-rim	-
5	0.44	97.26	0.81	0.00	0.04	1.37	0.00	0.00	0.06	99.98	Si-Core	-
6	92.66	4.48	0.88	0.00	0.02	0.00	0.55	0.33	1.08	100.00	Ca-rim	-
7	1.22	51.42	20.90	0.24	24.96	0.00	0.03	0.12	1.10	99.99	Mg-Al-Silicate	-
8	0.42	57.38	16.04	0.00	24.03	0.00	0.08	0.07	1.99	100.01	Mg-Al-Silicate	-
9	5.84	44.00	21.79	0.00	27.97	0.00	0.09	0.00	0.29	99.98	Mg-Al-Silicate	-
10	1.00	52.16	17.34	0.00	26.58	0.00	0.00	0.10	2.81	99.99	Mg-Al-Silicate	-
11	71.86	10.97	0.57	0.20	8.78	0.36	0.32	0.54	6.40	100.00	Ca-Mg-Si-Fe-Al Interstitial Phase	0.43
12	66.24	10.00	5.47	0.00	11.23	0.00	0.33	0.36	6.36	99.99	Ca-Mg-Si-Fe-Al Interstitial Phase	0.47
13	69.16	10.15	5.04	0.02	10.64	0.00	0.43	0.33	4.22	99.99	Ca-Mg-Si-Fe-Al Interstitial Phase	0.44
14	59.71	16.06	6.81	0.11	11.73	0.26	0.45	0.92	3.95	100.00	Ca-Mg-Si-Fe-Al Interstitial Phase	0.73
15	9.35	16.40	0.98	0.00	0.85	0.00	0.46	0.27	71.68	99.99	Fe-Oxide	-
16	1.30	49.05	20.97	0.00	24.17	0.00	0.14	0.00	4.36	99.99	Mg-Al-Silicate	-



X-ray Diffraction – Mineralogical Composition of Mortar

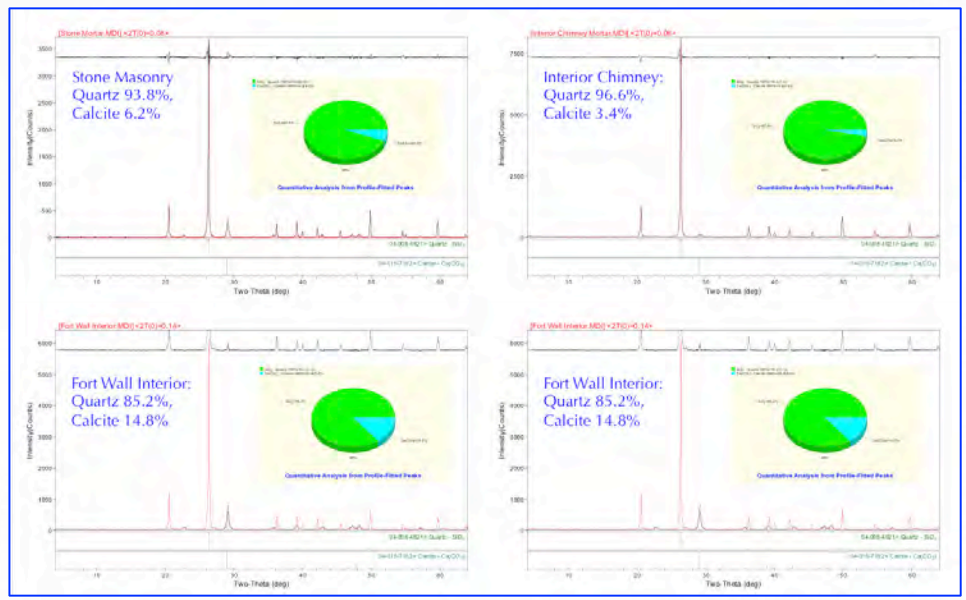


X-ray Diffraction of Mortars From Fort Washington

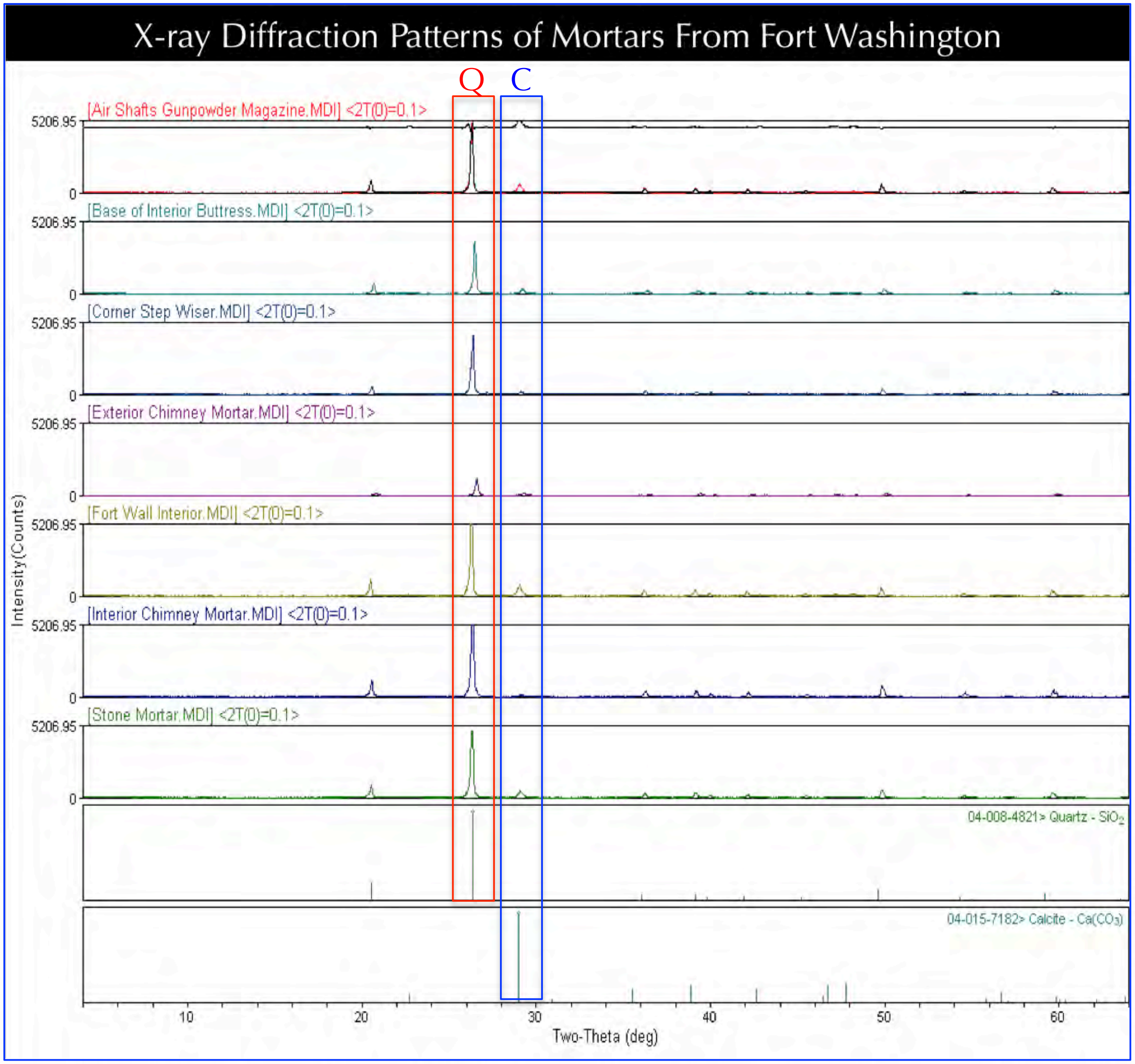


Qualitative and Semi-Quantitative Estimates of Minerals From

- Sand
- Binder
- Salts
- Pigments

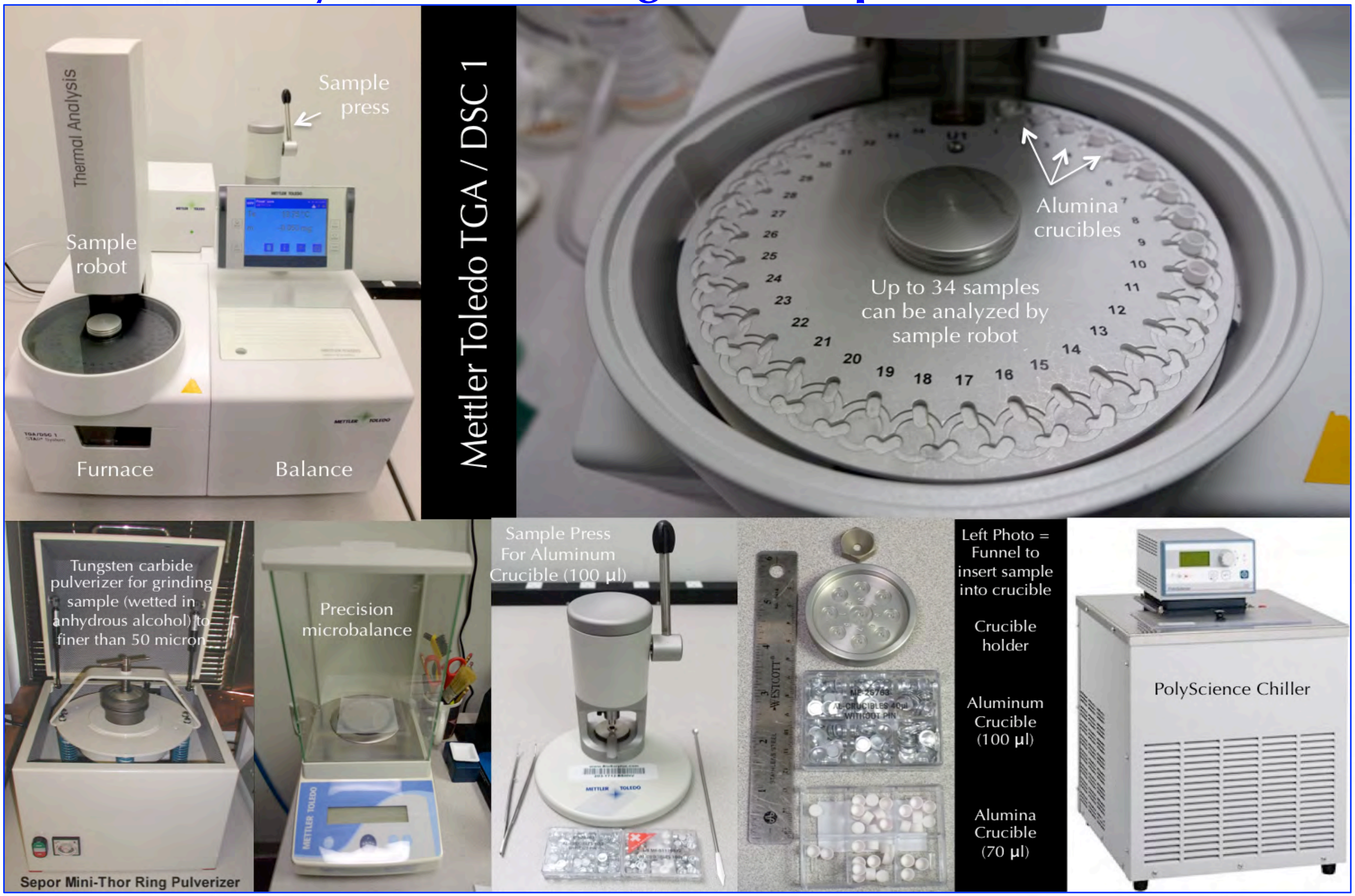


Relative Proportions Of Quartz and Calcite In Different Mortars From Fort Washington



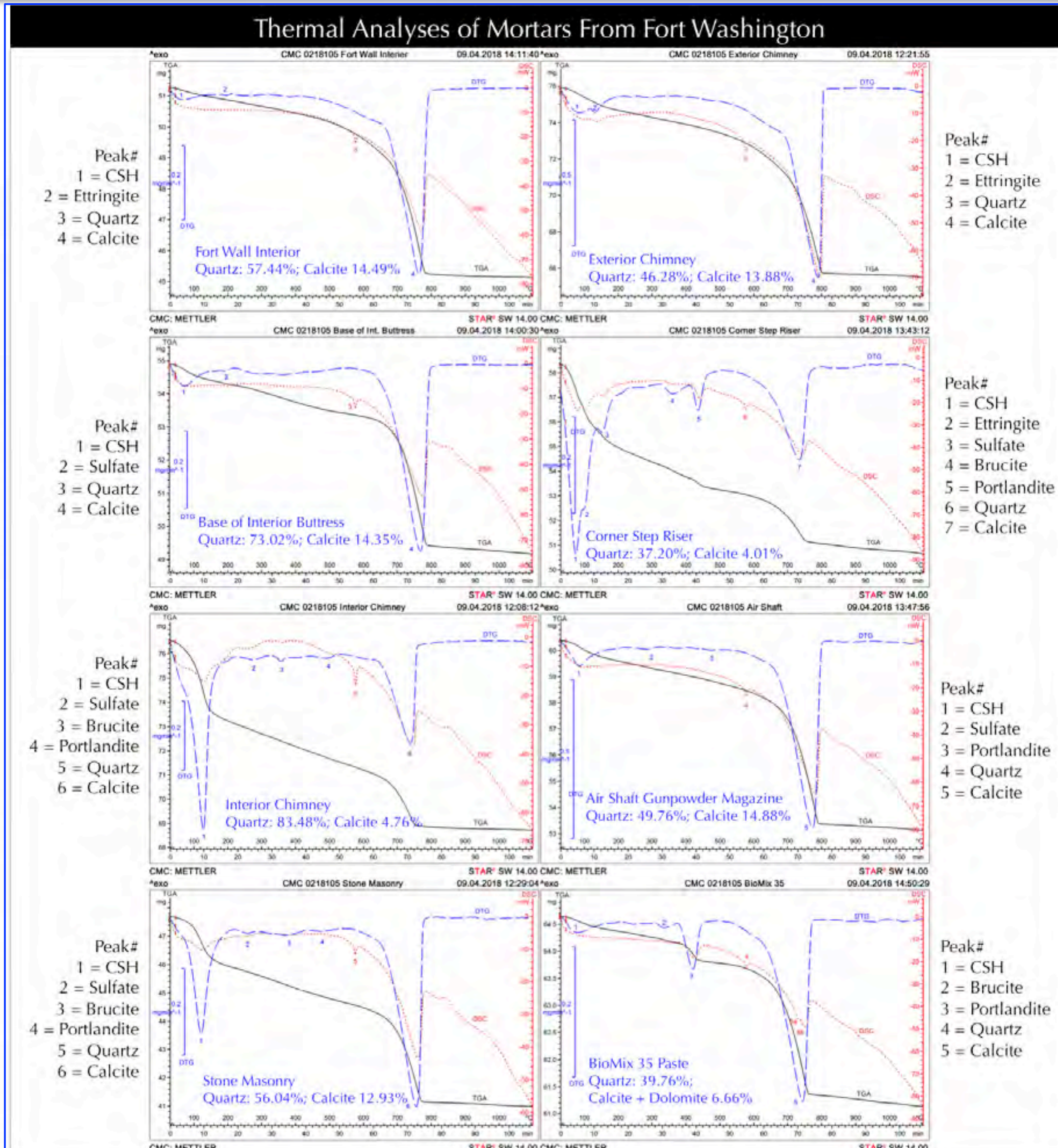
X-ray Fluorescence – Chemical Composition of Mortar

Thermal Analysis – Mineralogical Composition of Mortar

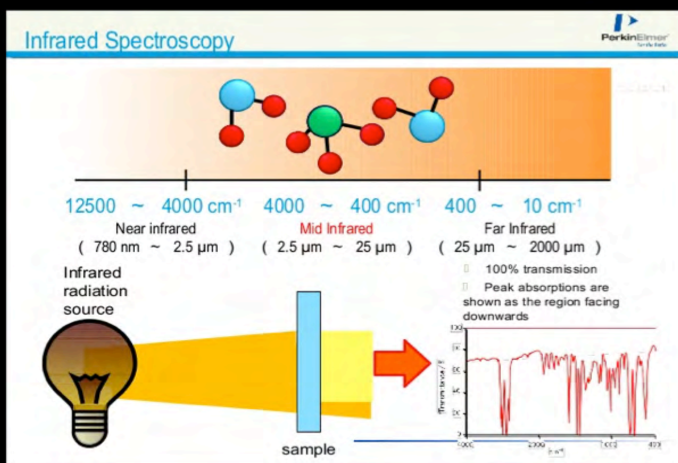


Thermal Analyses Of Mortars From Fort Washington

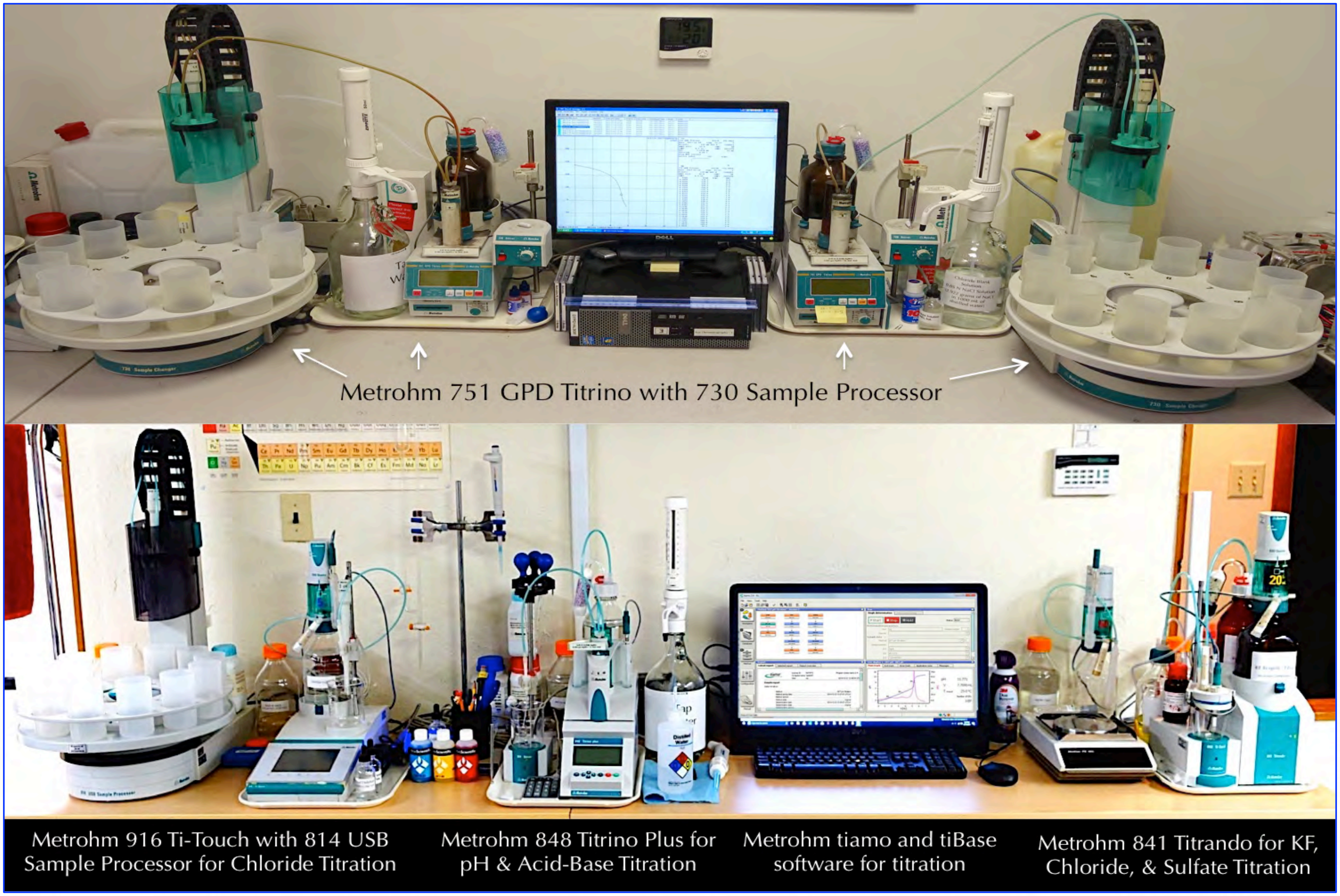
- Lime mortars from Fort Wall, Interior Buttress have main peaks for Calcite and Quartz
- Altered Lime mortars from Interior Chimney, and Stone Masonry have main peaks for Hydrate water along with Calcite and Quartz
- Slag Cement – Lime mortar from Exterior Chimney has main peaks for Calcite and Quartz
- Blended Cement – Lime mortar from Corner Step Riser Terri Plane has most complex endotherms
- Masonry Cement mortar from Air Shafts Gunpowder has main peaks for Calcite and Quartz
- BioMix 35 has main peaks for Brucite, Portlandite, Calcite, Dolomite, and Quartz



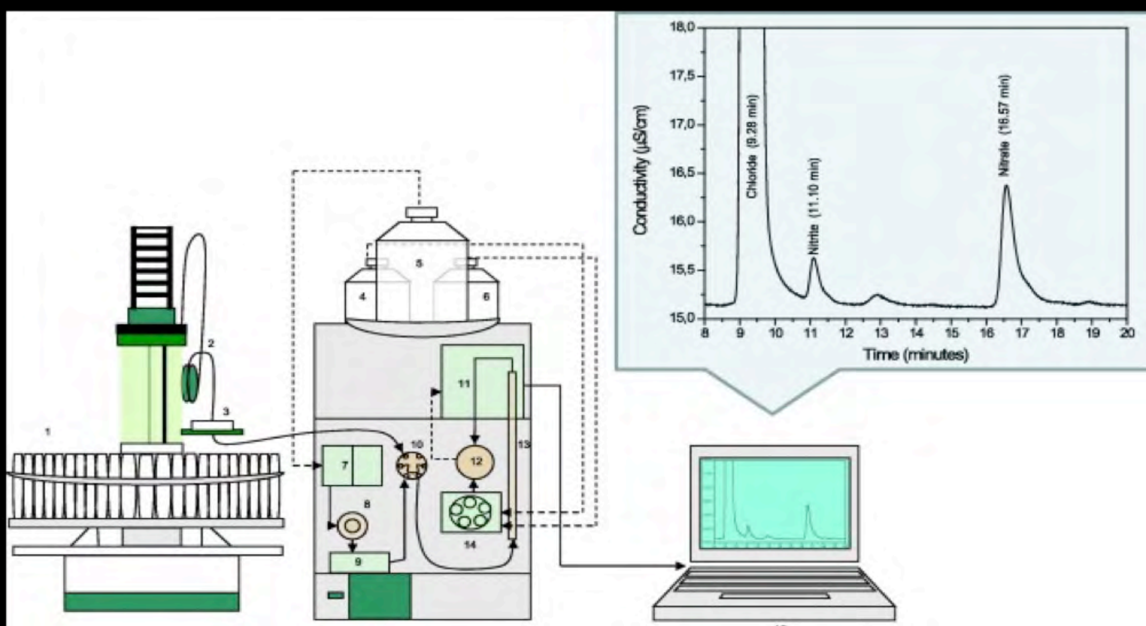
Infrared Spectroscopy

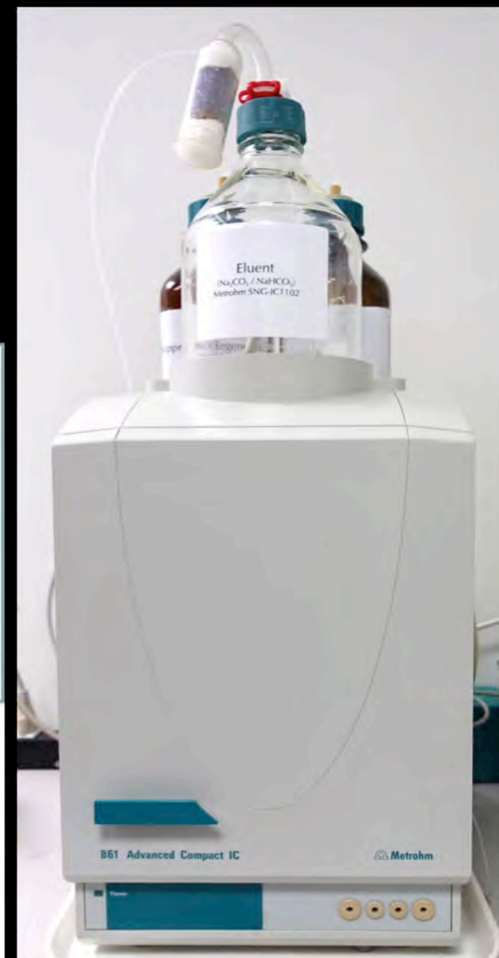


Chloride Analysis

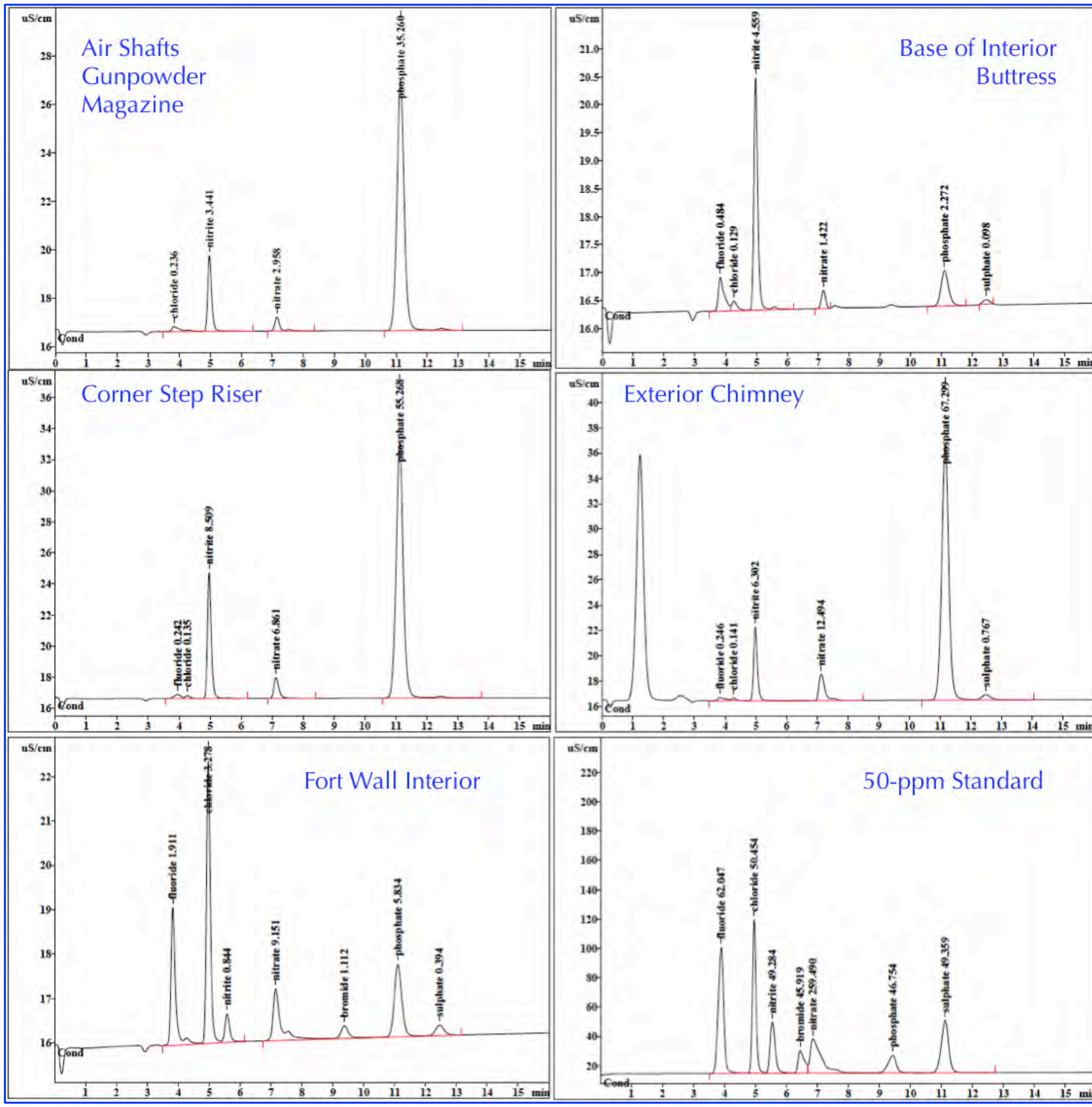


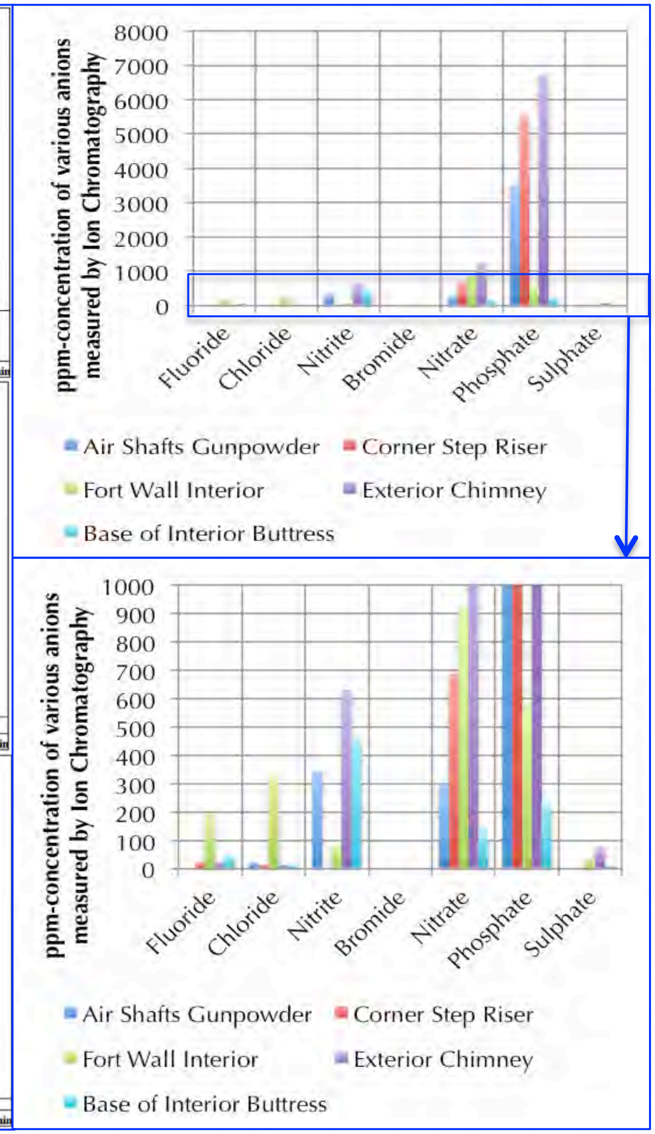
Ion Chromatography
(Chloride, Sulfate, Nitrate, Phosphate Salts)





Water-Soluble Salts in Fort Washington Mortars From Ion Chromatography





Making Sense From XRF - Chemical - XRD – Thermal Data

Data	Method	1	2	3	4	5	6	7	8	9	10	11	12	13	14
SiO_2	XRF	69.1	75.5	74.8	65.2	68.6	57.3	65.1	62.2	67.7	63.8	51.2	69.5	78.4	1.24
Al_2O_3	XRF	1.65	2.3	2.22	1.45	1.61	3.46	2.84	6.16	6.89	1.55	1.91	4.22	1.72	0.913
Fe_2O_3	XRF	1.43	2.18	1.49	1.37	1.75	2.13	2.19	2.14	2.15	2.26	3.07	1.35	1.01	1.16
CaO	XRF	14.4	9.33	4.49	11.8	14.5	17.8	14.1	14.1	12.3	12.0	13.6	12.2	9.84	36.7
MgO	XRF	1.38	1.72	3.06	3.31	1.47	1.44	1.23	4.02	1.26	0.637	1.48	2.81	3.77	ND
Na_2O	XRF	ND	ND	ND	ND	ND	ND	ND	1.25	1.4	ND	ND	2.85	0.093	ND
K_2O	XRF	0.507	0.588	0.111	0.099	0.239	0.738	0.865	2.06	2.27	0.315	0.56	0.917	0.101	0.598
TiO_2	XRF	0.188	0.239	0.114	0.127	0.37	0.604	0.578	0.2	0.155	0.435	0.955	0.112	ND	0.094
P_2O_5	XRF	0.046	0.127	0.103	0.042	0.049	0.081	0.101	0.081	0.087	0.026	0.012	0.292	0.044	0.226
SO_3	XRF	ND	ND	ND	ND	0.907	0.456	0.1	0.625	ND	4.55	6.63	0.298	0.038	0.489
Balance	XRF	11.6	8.34	14	16.9	10.5	16	12.8	7.16	6.06	14.4	20.5	5.42	4.95	57.8
LOI @ 110°C	Wet	4.0	1.8	2.7	4.6	2.7	8.9	4.5	0.6	-	0.39	1.90	-	-	4.8
550°C		3.4	2.8	4.9	5.4	2.4	3.7	2.2	2.8	-	2.00	4.10	-	-	8.6
950°C		8.6	6.1	3.8	8.0	10.1	3.9	8.0	5.2	-	7.20	7.40	-	-	28.9
Acid-Insoluble Residue (%)	Wet	68.6	74.7	76.3	64.3	63.9	57.8	66.8	65.5	-	74.4	62.5	-	-	0.67
		52.4	53.9	-	-	-	53.7	61.8	-	-			-	-	-
Soluble Silica, wt.%	Wet	3.33	2.13	8.55	7.54	9.21	11.67	7.37	-	-			-	-	-
Quartz	XRD	85.2	88.5	96.6	93.8	82.5	91.7	83.0	-	-	66.9	63.2	-	-	2.8
	TGA/DSC	57.4	73.0	83.5	56.0	46.3	37.2	49.7	-	-	-	-	-	-	-
Calcite	XRD	14.8	11.5	3.4	6.2	17.5	8.3	17.0	-	-	15.7	29.5	-	-	26.2
	TGA/DSC	14.5	14.3	4.7	12.9	13.8	4.0	14.8	-	-	-	-	-	-	32.4

Samples 1–4: Original (lime) mortars of Fort Washington
Samples 5–7: Pointing (cement) mortars of Fort Washington
8: Rosendale 12M
9: BioMix 35
10: Historic Non-hydraulic Lime mortar
11: Historic NC-Lime mortar
12: Fort Sumter
13: Fort Moultrie
14: Fort Zachary Taylor

Mix Calculations – From Petrography, Chemical, Thermal Data

Steps Followed During Laboratory Testing of Hardened Mortar

STEP 1

Visual Examinations
Photographs
Mortar Petrography
- Sand Type
- Sand Mineralogy, Microstructure
- Sand grain-size distribution
- Sand size, shape, color, gradation, distribution, soundness, reactivity
- Binder Mineralogy
- Binder Type(s)
- Mortar Type

All from
- Optical Microscopy
- SEM-EDS
- XRD

→

STEP 2

Mortar Chemistry
- Acid-digestion for acid-insoluble residue content (siliceous component of sand; silt and clay residue in a mortar with calcareous sand)
- Wet chemistry (Cold-HCl and hot-NaOH digestion) followed by instrumental analysis (XRF, AAS, ICP) for soluble silica content of mortar
- Instrumental analysis (XRF, AAS, or ICP) for bulk oxide compositions of mortar
- Free and Combined Water, & Carbonation from Losses on Ignition

→

STEP 3

Mortar Mineralogy from
- X-ray diffraction (for sand content, salts, clay, secondary deposits, binder mineralogy, etc.
- Thermal analysis (hydrous phases, CSH, sulfates, hydroxides, e.g., brucite, portlandite, carbonates, e.g., fine-grained carbonated paste, coarse calcite, dolomite, etc.)

→

STEP 4

Methods of calculations of relative volumes of sand and binders

Assumed Compositions of the Binder Components
- Lime
- Historic Binder
- Portland Cement
- Masonry Cement
- Slag Cement
- Natural Cement

Assumed Densities of Mortar Ingredients

→

STEP 5

Calculations of mix proportions of mortar from determined compositions of mortar and assumed compositions and densities of sand and binder

- Non-hydraulic Lime : Sand
- Hydraulic Lime : Sand
- Natural Cement : Sand
- Natural Cement : Lime : Sand
- Portland Cement : Lime : Sand
- Masonry Cement : Sand
- Final Mortar Type

Petrography → Chemistry → XRD, Thermal → Assumptions → Mix Calculations

Mix Calculations Of Modern Masonry Mortars

Problems in Mix Calculations of Historic Mortars

- Alterations
- Unknown Binder Composition
- Pozzolans

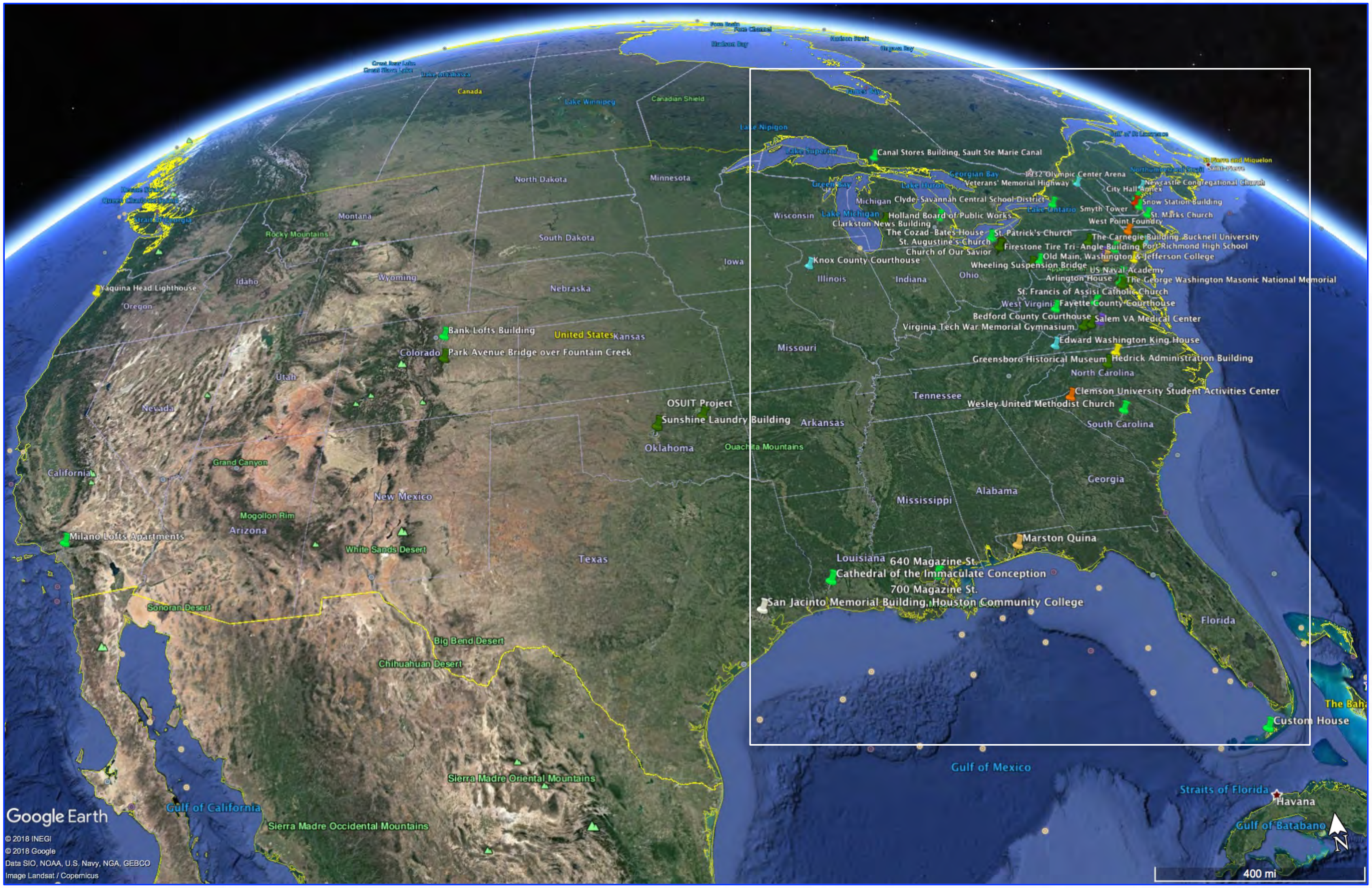
Binders and Sand	Assumed Compositions and Methods of Calculation	Assumed Bulk Density (lbs./ft^3)
High-Calcium Non-hydraulic Lime	[CO_2 data from loss on ignition at 950°C divided by 0.594], where 0.594 is ratio of molecular weights of CO_2 to $Ca(OH)_2$ i.e. 44/74.09	40
Magnesian Non-hydraulic Lime	[100 times (brucite content in mortar from TGA/DSC/5.8)], assuming magnesian lime has 71% CaO and 4% MgO, or 5.8% brucite, since ratio of molecular weights of brucite to MgO (58.32 / 40.32) is 1.447	40
Dolomitic Non-hydraulic Lime	[100 times (brucite content in mortar from TGA or DSC divided by 42)], assuming dolomitic lime has 41% CaO and 29% MgO, or 42% brucite, since ratio of molecular weights of brucite to MgO (58.32 / 40.32) is 1.447	40
Calcitic or Magnesian Hydraulic Lime	[100 times (soluble silica in mortar/0.07] assuming hydraulic lime has 7% SiO_2 , or average SiO_2 content calculated from SEM-EDS data of paste	40
Dolomitic Hydraulic Lime	[100 times (soluble silica in mortar/0.07] assuming hydraulic lime has 7% SiO_2 , or average SiO_2 content calculated from SEM-EDS data of paste Or [100 times (brucite content in mortar from TGA/DSC/38)], assuming lime has 38% CaO and 26% MgO, or 38% brucite, since ratio of molecular weights of brucite to MgO (58.32 / 40.32) is 1.447	40
Portland Cement in Cement-Lime Mortar	100 × [Soluble silica in mortar / 21.0], assuming 21% silica in Portland cement	94
Calcitic Lime in Portland Cement-Lime Mortar	Lime content = 1.322 × CaO assignable to Lime, which is [CaO content of Mortar – (CaO assignable to portland cement, which is portland cement content × 0.635, assuming 63.5% CaO in portland cement)], where the factor 1.322 comes from ratio of molecular weights of $Ca(OH)_2$ to CaO i.e. 74.09/56.03	40
Dolomitic Lime in Portland Cement-Lime Mortar	100 times (brucite content in mortar from TGA/DSC/42)], assuming dolomitic lime has 41% CaO and 29% MgO, or 42% brucite, since ratio of molecular weights of brucite to MgO (58.32 / 40.32) is 1.447	40
Slag Cement	100 × [Soluble silica in mortar / 27.0], assuming 27% silica in slag cement, or average SiO_2 content determined from SEM-EDS data	90
Natural Cement	100 × [Soluble silica in mortar / 20.0], assuming 20% silica in natural cement, or average SiO_2 content determined from SEM-EDS data	75
Masonry Cement	(i) 100 – [Sand + Total Water], if sand is all siliceous and hence sand content is obtained directly from the acid-insoluble residue content; (ii) PC content (from the soluble silica data) divided by factor 0.50, 0.66, or 0.75 with an assumed masonry cement type of N, S, or M, respectively. MC Type (M, S, N) is determined from PC/MC = 0.75 (for M), 0.66 (for S), or 0.50(for N) – if sand has calcareous component	70 (Type N) 75 (Type S) 80 (Type M)
Gypsum Plaster	Gypsum content from XRD or thermal analysis times 0.843 (ratio of molecular weight of plaster to gypsum)	70
Sand	If sand contains acid-soluble component (carbonates), Sand content = 100 – [Total Binder + Total Water from LOI to 550°C i.e. free plus hydrated water]; If sand has no acid-soluble component (i.e. all siliceous sand) Sand content is directly obtained from the acid-insoluble residue content	80

Mix Calculations

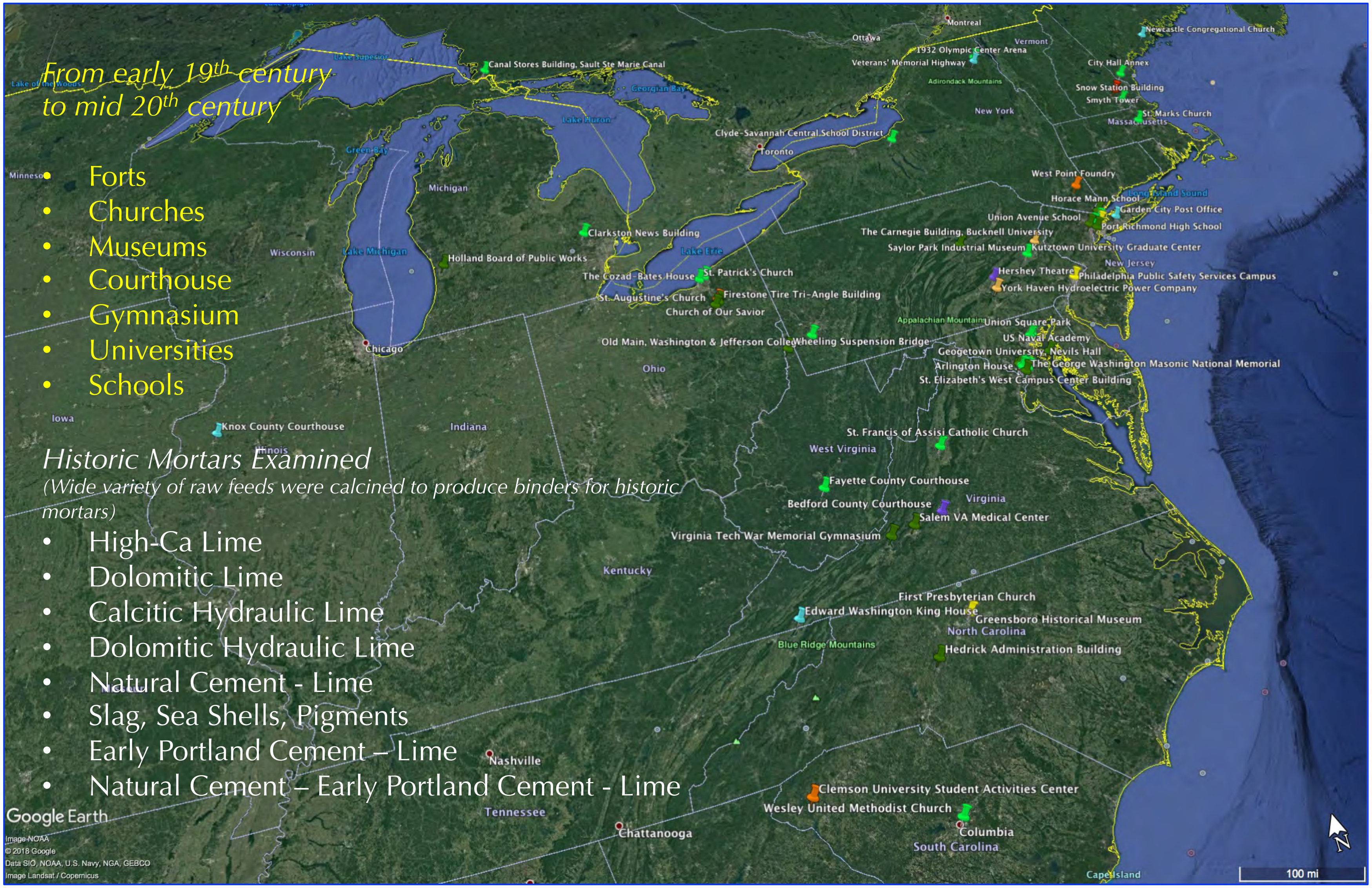
Locations	Fort Wall Interior	Base of Interior Buttress	Interior Chimney	Stone Masonry
Mortar Type	Least Altered *Hydraulic Lime – Silica Sand Mortar*	Moderately Altered *Hydraulic Lime – Silica Sand Mortar*	Highly altered *Hydraulic Lime – Silica Sand Mortar*	Highly altered *Hydraulic Lime – Silica Sand Mortar*
Calculated Hydraulic Lime: Sand, by volume	1-part lime to 2.2-part sand	1-part lime to 2.0-part sand	1-part lime to 1.8-part sand	1-part lime to 1.5-part sand
Proposed Lime-to-Sand Proportions, by Volume	***1-part hydraulic lime to 2-part sand, by volume***			
Suggested Pointing Mortars	BioMix 35 or 50 (Std. White to Std. Buff variety to try and match), or, 1-part BioLime NHL 3.5 (Buff to White variety to mix and match) to 2 to 2.5-part silica sand			

Locations	Exterior Chimney	Corner Step Riser Terri Plane	Air Shafts Gunpowder Magazine
Mortar Type	Slag Cement – Lime – Silica Sand Mortar	Blended Cement (Portland cement, Fly Ash) – Lime – Limestone fines – Silica Sand Mortar	Masonry Cement (Portland cement, lime, limestone fines) – Silica Sand Mortar
Calculated Binder: Sand, by volume	1 : 2.3	1 : 2.2	1 : 2.2
Proposed Binder-to-Sand Proportions, by Volume	***1-part binder to 2½-part sand, by volume***		
Suggested Repointing Mortars	Slag cement – Lime Mortar; ASTM C 270 Type N or S Cement-Lime Mortar; Natural Cement-Lime Mortar	ASTM C 270 Type S Cement-Lime Mortar	ASTM C 270 Type N Masonry Cement Mortar

CMC's Historic Mortar Testing from East to West Coasts



CMC's Historic Mortar Testing From the East Coast



From early 19th century to mid 20th century

- Forts
- Churches
- Museums
- Courthouse
- Gymnasium
- Universities
- Schools

Historic Mortars Examined

(Wide variety of raw feeds were calcined to produce binders for historic mortars)

- High-Ca Lime
- Dolomitic Lime
- Calcitic Hydraulic Lime
- Dolomitic Hydraulic Lime
- Natural Cement - Lime
- Slag, Sea Shells, Pigments
- Early Portland Cement – Lime
- Natural Cement – Early Portland Cement - Lime

CMC's Historic Mortar Testing From Various Forts

Fort Sumter (Charleston, SC)
Natural cement concrete
Natural cement – lime mortar, circa 1850

Fort Mott (Pennsville, NJ)
Natural cement – lime mortar, circa 1872

Fort Jackson (Columbia, SC)
Masonry cement pointing mortar, circa 2009

Fort Moultrie (Sullivan's Island, SC)
Dolomitic lime mortar, circa 1812

Fort Zachary Taylor East (Key West, FL)
Natural cement – lime - beach sand mortar, circa 1845

Fort Washington (Maryland)
Hydraulic lime mortar, circa early 1800s

Think CMC For your next Laboratory Testing Of Historic Mortars

"CMC employs an impressive array of technological tools to provide comprehensive analyses for historic masonry mortars. They also deliver results on a reliable schedule to meet the time pressure of our clients' projects."

Mike Edison
Edison Coatings, Inc.

"CMC, Inc. is an excellent resource for petrographic analysis of historic mortars. Mortar analysis for historic buildings must go beyond just understanding the components of the mortar to understanding how those components are contributing to the success or failure of that mortar over time. This is where CMC, Inc. succeeds in guiding architects towards the right restoration mortar mix. The reports are thorough yet concise with reliable turn-around times. Dr. Jana is always willing to discuss the findings and his clients can count on his expertise."

Amanda Edwards
Sr. Architectural Conservator

Acid Digestion ($350-500)

$2000

Comprehensive Analysis ($1500-$2750)