Project: USHG #10027

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Michael Trinkley
Chicora Foundation, Inc.
PO Box 8664
Columbia, SC 29202
Phone: 803-787-6910

EVALUATION OF MORTAR COMPOSITION – ASTM C1324
DADE PYRAMIDS, ST. AUGUSTINE NATIONAL CEMETERY

1.0 INTRODUCTION

We are pleased to present the results of our laboratory testing of sample of mortar removed from the "cement" base of coquina stone pyramids at St. Augustine National Cemetery. We understand that this section of the structure was originally constructed in 1842 and is currently undergoing renovations.

2.0 METHODOLOGY

ASTM C1084 - The sample was analyzed for cement content according to chemical procedures and petrographic examination methods of ASTM C1084, "Standard Test Method for Portland Cement Content of Hardened Hydraulic-Cement Concrete".
ASTM C856 - The sample was examined according to methods of ASTM C856, .Standard Practice for Petrographic Examination of Hardened Concrete. using a stereomicroscope up to a magnification of 100X. Portions of the binder portion of the concrete were prepared on glass slides in several refractive index oils in the range of 1.30 to 1.71 and examined for identification using a polarizing (petrographic) microscope up to a magnification of 1000X. The optical and morphological properties of the phases present were used to identify the various constituents present, including primary and secondary calcium carbonate, hydrated lime, gypsum, brucite, free lime, portland cement, and any other substances. The sample was also examined using Electron Microscopy.
Chemical Analysis - The chemical analysis was conducted, using wet chemical procedures in ASTM C1324 and C1084, and X-ray fluorescence spectroscopy (XRF), and thermal analysis.

3.0 RESULTS

3.1 PETROGRAPHIC EXAMINATION

General Observations - The sample fragments appear to consist of hardened concrete containing natural cement, natural sand fine aggregate and coarse aggregate.

Paste Properties - The paste has a light-to-medium gray color. The paste consists of carbonated hydrated lime and hydrated natural cement. The paste is hard, carbonated, and has good paste-aggregate bond, with good firmness. Brick fragments are not present on sample surfaces. The degree of hydration is highly advanced. The fine aggregate volume appears moderately high. Pockets of carbonated hydrated lime, up to 4 mm in size, are common. This concrete appears to have been made from a hydraulic hydrated lime and natural cement. This sample consists of concrete (not mortar).

Aggregate - The coarse aggregate consists of 0.8 inch maximum observed size coarse aggregate consisting of white fine-grained dolomite and quartz. The fine aggregate is a natural sand consisting predominantly of quartz and feldspar, with lower amounts of limestone, dolomite, basalt, and hornblende. The aggregates appear to be in a physically and chemically stable condition. Approximately 44% of the aggregate volume is coarse aggregate, and 56% is fine aggregate (sand).

Air Content - The concrete air content is 1.1%. The air-voids are entrapped, with sizes greater than 250 microns. The sample is not air-entrained.

3.2 CHEMICAL ANALYSIS

The cement content, including portland or natural cement, was determined according to ASTM C1084, "Standard Test Method for Portland Cement Content of Hardened Hydraulic-Cement Concrete". The analysis was conducted according to the Maleic Acid procedure. This method extracts all of the hydrated cement compounds, excluding the carbonate rock aggregate and carbonated hydrated lime. It does not extract any of the aggregates present in this sample. Natural cement is similar to portland cement, however it is produced from a single raw material. It usually has lower sulfur trioxide (SO_3) and higher magnesium oxide (MgO) contents than modern-day portland cements.

The paste portion was analyzed for SO_3, MgO and CaO (calcium oxide), using X-ray Fluorescence (XRF).

The portland/natural cement was estimated to contain 63.5% CaO and 21.0% SiO_2 (silicon dioxide). The hydrated lime content was estimated based on the amount of brucite ($Mg(OH)_2$) and lime (CaO). The hydrated lime was estimated to contain 43.0% CaO and 29.0% MgO, which is equal to 42.0% $Mg(OH)_2$.

Based upon microscopy, and chemical analysis of the paste, the binder portion of this sample was found to consist of natural cement and hydrated lime. The aggregate content was calculated as the insoluble residue minus the carbonated hydrated lime. The hydrated lime appears to be a dolomitic type. The hydrated lime content was calculated based upon the brucite content in the mortar fraction of the concrete. The natural cement was assumed to contain 63.5% calcium oxide (CaO) and 21.0% silicon dioxide (SiO_2). The amount of natural cement is based on the quantity of extract in the maleic acid method.

The densities (loose volume basis) of the sample ingredients were assumed to be those listed in ASTM C270. Eighty lbs. of dry sand was assumed to be equal to one cubic foot of damp loose sand. The bulk density of the hydrated lime was assumed to be 40 lbs./ft_3 and the bulk density of the natural cement was assumed to be 94 lbs./ft^3.

The volumetric proportions of the sample (determined according to ASTM C270) are as follows:

Constituents:	St. Augustine Natl. Cemetery
Natural Cement	604 lbs./yd.^{3}
Hydrated Lime	34 lbs./yd.^{3}
Total Aggregate	3016 lbs./yd.^{3} (approx. 44% is Coarse Agg., and 56% is Sand)
Total Water	290 lbs./yd.^{3}
Concrete Density	3944 lbs./yd.^{3} (S.S.D.) Saturated
Water-Cement Ratio	0.480
Water/Total Binder Ratio	0.455

At the time that this concrete was reported to have been cast (circa 1842) the major dominant cement in the USA was natural cement. Based upon the higher than normal MgO content, and lower SO_3 content relative to the portland cement content, this concrete sample appears to have been made with natural cement.

The microstructure of the paste is most similar to natural cement. (Refer to attached photographs, which show residual rhombic crystals of calcined limestone and calcined dolomite, which is a constituent in hydrated natural cement; These crystals have carbonated with age, and are not found in hydrated portland cement paste). Joseph Aspidin acquired his patent on portland cement in 1824-25. Imports of portland cement in 1842 were low. The earliest natural cement production in the USA occurred circa 1820 in: Lehigh Valley, Pa;Coplay, PA; Dixon, IL; and Ulster County, NY. The first American portland cement plant was reported to have begun operating in 1875, and by 1890 approximately seventeen plants in the USA were producing portland cement.

ASTM C219 defines Natural Cement, and ASTM C10 is the Specification for Natural Cement. Concrete made with the old natural cement from the 1800.s had longer set time, lower strength and lower modulus of elasticity than portland cement concrete and mortars. A modern form of a natural cement (which would be a portland cement produced from a single component, such as a very impure limestone) could approach the performance of a true portland cement.

The old form of natural cement had to be clinkered at a much lower temperature than portland cement, due to its high MgO content. A modern form of it could be clinkered (fired) at the same temperature as portland cement, provided the MgO is not high.

A summary of the chemical analysis results of the mortar fraction of the concrete is given in the attached Table 1.

Respectfully submitted,

U.S. Heritage Group, Inc.

Tom Glab
Laboratory Manager

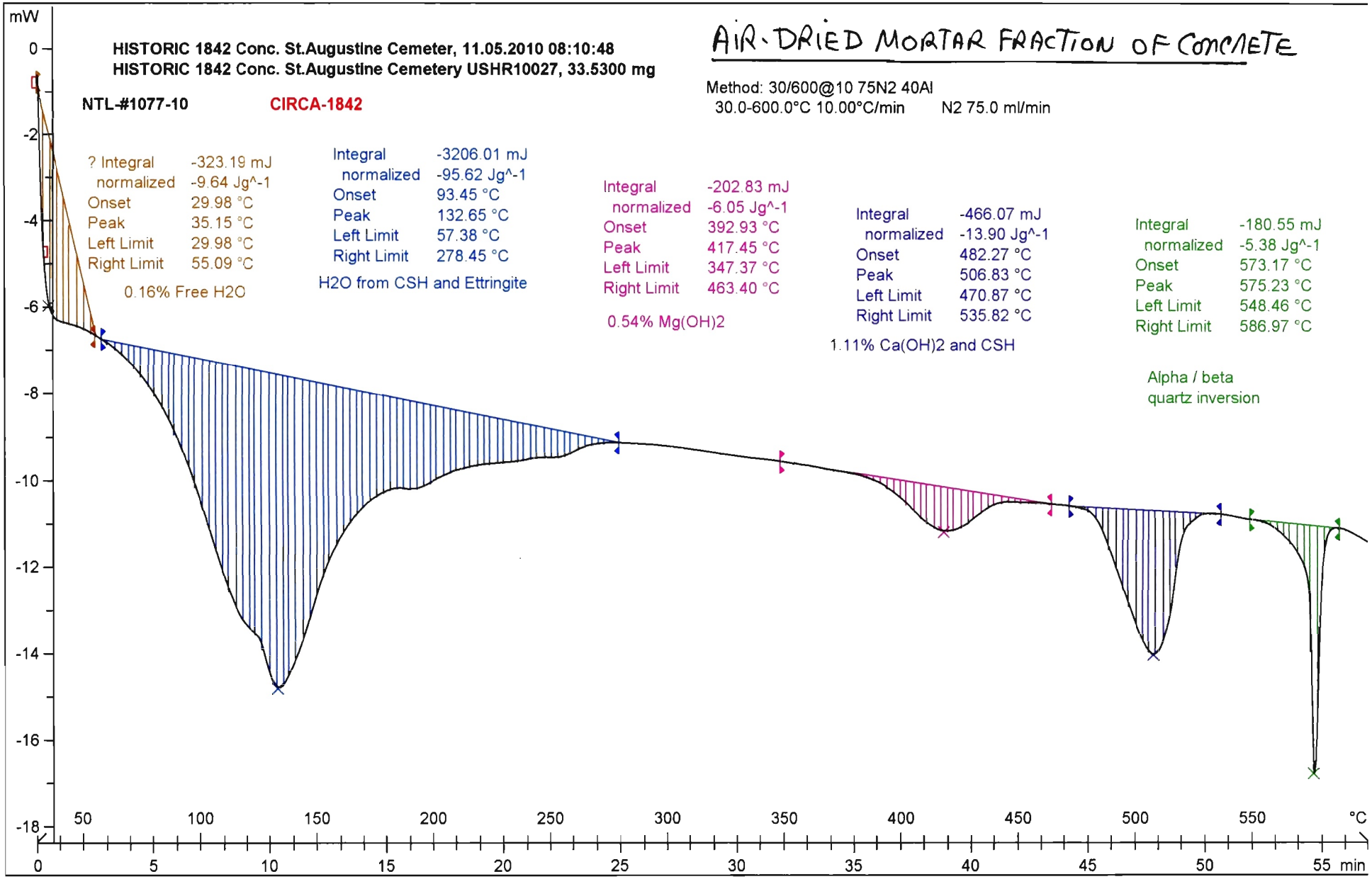
Nelson Testing Laboratories

Michael F. Pistilli, FACI
Chemist, Petrographer

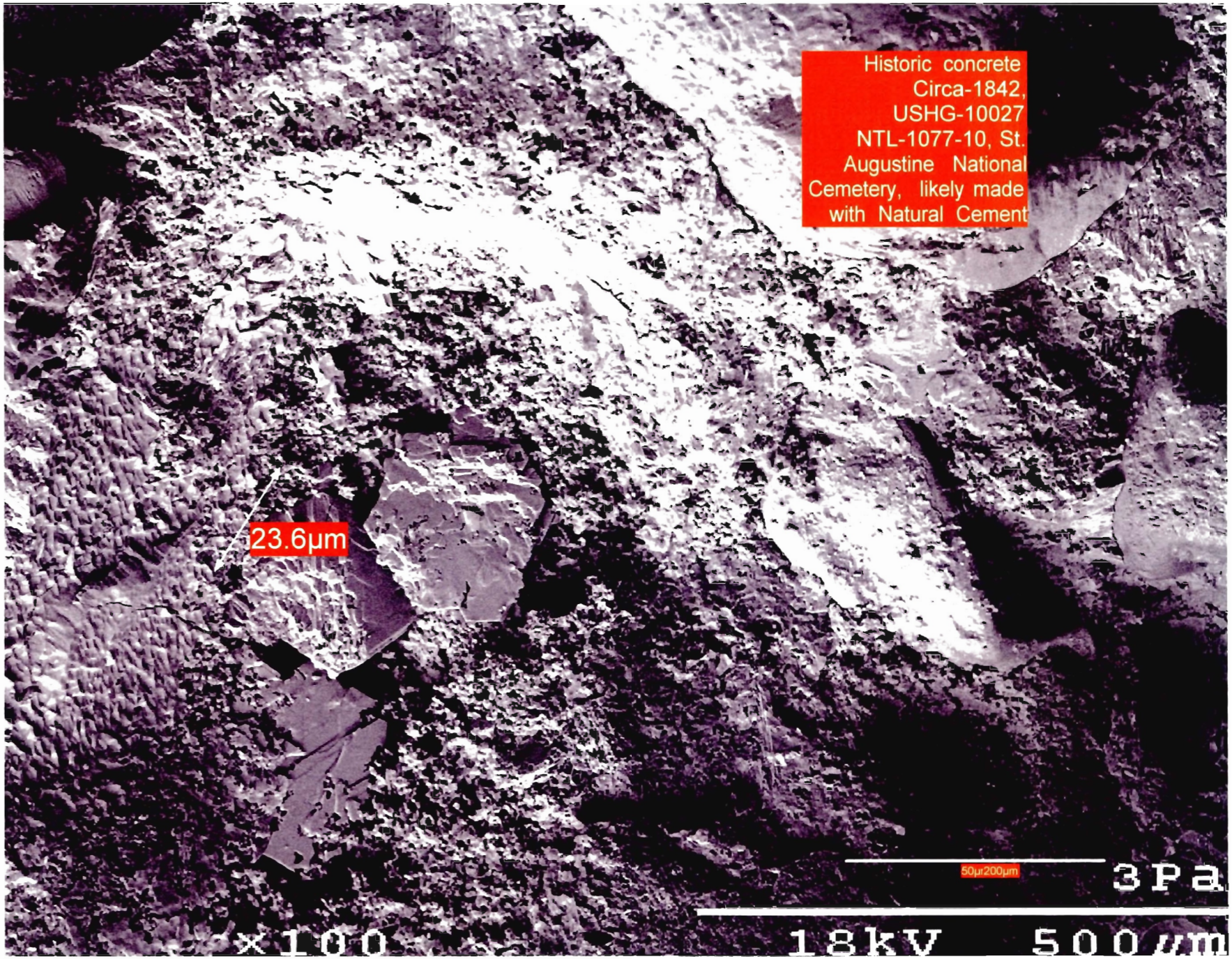
Table 1. Chemical Analysis of Mortar Samples

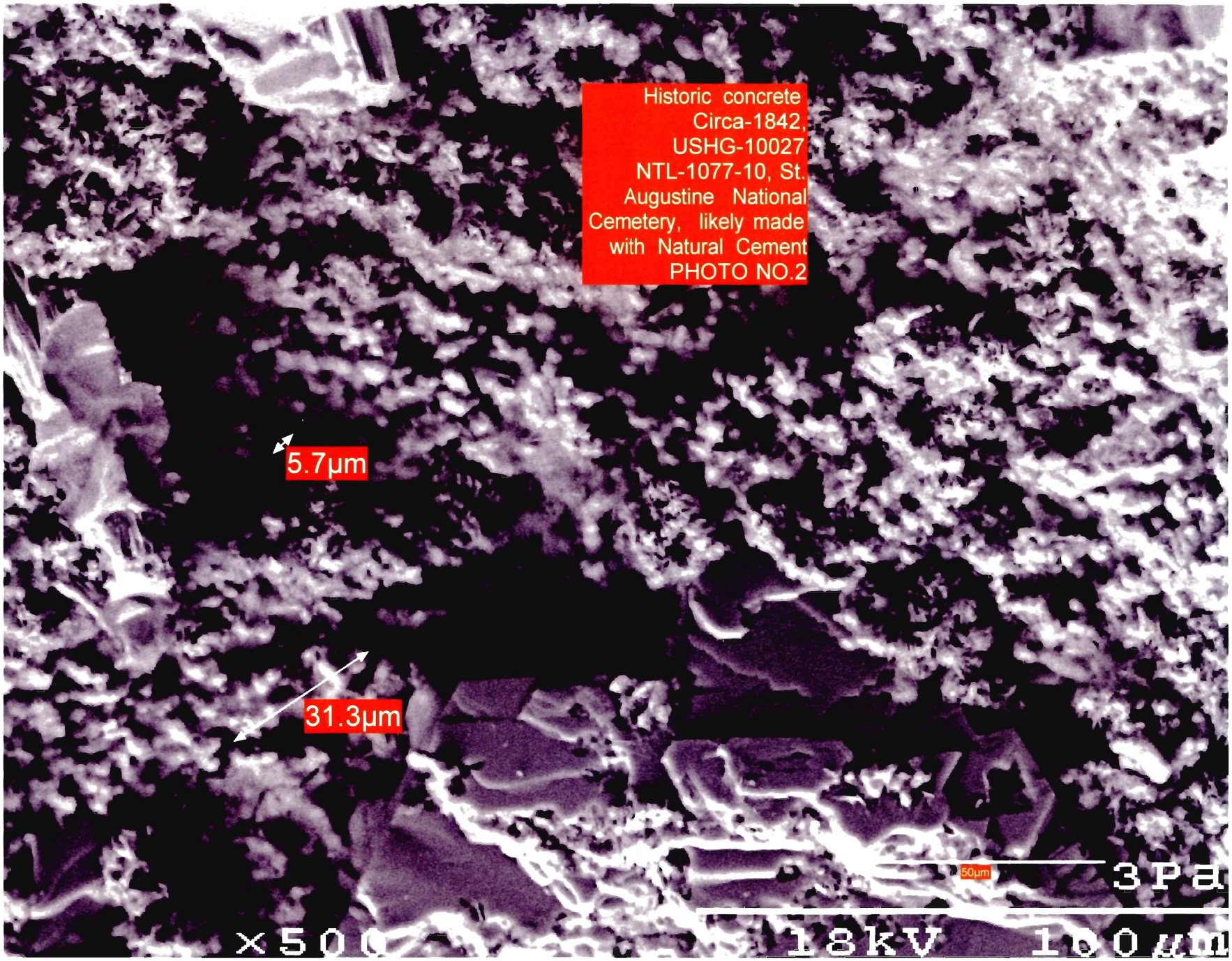
Constituent	Percent by Mass %
	Sample #1 St. Augustine Natl. Cemetery
Silica - SolubleSiO_2	Not Determined
Calcium Oxide - CaO	30.26
Brucite – $Mg(OH)_2$	0.54
Insoluble Residue	69.93
Magnesium Oxide – MgO	1.78
Sulfur Trioxide - (SO3)	0.47
Loss on Ignition	
At 0-110°C	0.0
At 110-550°C	4.40
AT 550-1000°C	3.15
Calculated Constituents	
Natural Cement	24.82
Hydraulic Hydrated Lime	1.4
Sand	69.39

^exo HISTORIC concrete St.Augustine Cemetery 11.05.2010 09:16:45



Lab: m_pistilli STARe SW 8.10


Historic concrete
Circa-1842,
USHG-10027
NTL-1077-10, St.
Augustine National
Cemetery, likely made
with Natural Cement
23.6µm
50µm200µm
3Pa
x100
18kV
500µm


Historic concrete
Circa-1842,
USHG-10027
NTL-1077-10, St.
Augustine National
Cemetery, likely made
with Natural Cement
PHOTO NO.2
5.7µm
31.3µm
50µm
3Pa
×500
18kV
100µm