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Report on the Examination and Analysis of Mortar Samples from Brick Masonry

Wayne County Courthouse Nebraska, USA

Prepared and Approved By

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1.0 Introduction

Further to receipt of an e-mail enquiry from Douglas Elting of Berggren Architects on the 18th January 2018, a batch of samples from the Wayne County Courthouse, Nebraska in the USA, was received in CMC's Stirling Laboratory on the 1st February 2018.

The samples were identified as mortar samples removed from a selection of locations, and included both pointing and bedding mortars, with, in addition, a sample of sandstone masonry and one complete brick with adhering mortar was also included.

We were advised that the building was constructed in 1899 and has over the years undergone various forms of repair to the pointing mortars, employing a range of, not necessarily appropriate, more modern materials. Details of the locations sampled was supplied via email on the 24th January 2018, in the form of a selection of digital images showing the location of the masonry sampled.

On receipt the samples were examined, and a laboratory programme of analysis was proposed, along with an indication of costs, see CMC quotation, reference M/Q579/18, dated the 18th February 2018. Instruction to proceed with the analysis was received by email from Douglas Elting of Berggren Architects, on the 28th February 2018.

This report details observations from the examination of the samples along with the results of analyses carried out, and concludes with comment on the composition of the mortars and the binder type used. In addition, the response to the questions posed by the client are also included.

2.0 Sample

A batch of samples was received in CMC's Stirling laboratory on the 1st February 2018, with the samples submitted by Berggren Architects. The samples being identified as masonry mortars from a selection of locations on the Wayne County Courthouse, Nebraska, USA.

On receipt in the laboratory the sample details were entered into the sample register and the unique sample reference SR2552 allocated. Details of the sample locations, and the laboratory and Client sample references, are reproduced below for reference:

CMC Sample Reference.	Client Sam Reference.	aple Location/Comment
SR2552-S1	Brick	Taken from top of masonry wall in the attic, original construction.
SR2552-S2	Brick	Fragment of brick & mortar from inside of observation level in Tower.
SR2552-S3	Sandstone	From foundation to assess whether source of pigment used in mortar.
SR2552-S4	Mortar	Large mortar sample from inside column, observation level in Tower.
SR2552-S5	Mortar	Various mortar samples from original mortar within Tower.
SR2552-S6	Mortar	Small sample of original brick mortar, North side of Structure.
SR2552-S7	Mortar	Repointing mortar from ledge on North side of structure.
SR2552-S8	Mortar	Sample of mortar from area above stone masonry, west Elevation.



A selection of digital images was forwarded from the Client, with these showing the location of each sample submitted. These are reproduced in Appendix "A" to this report, for reference purposes.

Image No	Sample reference
2241 & 2685	SR2552 - S4
2212 & 2211	SR2552 – S5
2652	SR2552 – S6
2147 & 2651	SR2552 - S7
2650	SR2552 – S8

In addition to the above plates No. 2656, 2658, 2661, 2679 & 2683 were provided to demonstrate the condition and form of the mortar joints, observed *in situ*.

3.0 Methods of Examination and Analysis

On receipt in the laboratory all samples were initially submitted to a microscopic examination employing a stereo-binocular microscope at magnifications up to x20, and points of note recorded. During this examination representative sub-samples were exposed to a selection of reagents, dilute acids and indicator solutions and the response observed.

Based on the observations from the microscopic analysis the samples were grouped into "like" types, with representative samples from each group prepared and submitted to the following laboratory programme:

A selection of sub-samples was prepared and submitted to analysis by X-ray Powder Diffraction (XRD), to aid identification of the binder type employed and to ascertain whether the mortars had been affected by any potentially disruptive reactions.

Samples that contained binders which were indicated to contain Portland Cement or be moderate to eminently hydraulic lime-based mixes were analysed, by the methods of BS 4551: 2005 + A1: 2010 + A2: 2013, with those found to be high calcium or feebly hydraulic limes analysed by acid digestion, following the procedures of the SLCT (Scottish Lime Centre Trust).

Where considered appropriate, and sufficient sample was available, petrographic thin sections were prepared. This would enable additional information to be obtained, relating to the fabric condition of the mortar, and the form in which the binder was used at the time of mixing. The sections prepared were submitted to a petrographic examination in the Polarised light microscope.

4.0 Macroscopic Examination

A summary of the properties of the samples, in the as received condition, is presented below:



CMC Sample No.	Client Ref/Image	Mass (gram)	Maximum Dimension (mm)	Comment
SR2552-S1	Brick	134.6	31.7 x 25.6 6 x 9.6	Intact brick with adhering mortar With the measurements relating only to mortar in the sample. Mortar is relatively soft and locally friable, and is possibly hydrate mix. Grey/White colour.
SR2552-S2	Mortar	3.7	16.4 x 16.1 x 5.4	Small pieces of mortar removed from brick fragment. A pink coloured firm and compact mortar, with lime inclusions.
SR2552-S3	Sandstone	167.8	115 x 97.0 x 19.1	Fragment of weathered sandstone.
SR2552-S4	2241	177.4	88.8 x 55.7 x 13.5	Several intact pieces of brick bedding mortar. Mortar is pink in colour, well compacted and firm, but could be broken under light to moderate finger pressure.
SR2552-S5	2212	516.3	60.7 x 29.2 x 19.7	A number of sub-samples (A to F) comprising a quantity of fines and small intact fragments of mortar, very similar, visually, to sample S4.
SR2552-S6	2652	4.0	16.1 x 13.9 x 10.1	Small sample of original pink coloured pointing mortar with lime inclusions, well compacted and firm.
SR2552-S7	2147	32.0	30.3 x 25.3 x 10.1	Sample of repointing mortar, pink in colour, compact and moderately hard, but easily powdered
SR2552-S8	2650	7.7	35.7 x 13.8 x 6.8	Small fragments of pointing mortar, pink colour, compact and moderately hard, similar to sample S6, contains small inclusions.

Based on the above and given the quantity in each of the samples available for analysis, within each group, the following analysis programme was followed:

Analysis by XRD on all samples to confirm binder type, with a fines rich sample of the sandstone sample (SR2552-S3) included to clarify the components present in the matrix. This resulted in a total of 8 No. samples being submitted to analysis.

Mix composition of samples by acid digestion was limited by the quantity available, this included samples S1, S4 and S5, i.e. in total 3 No. samples were analysed by this method.

Mix composition by BS 4551, samples 4, 5, 7 & 8 were analysed by this procedure with samples S4 & S5 analysed for comparative purposes, i.e. a total of 4 No. samples.

In addition, a thin section was prepared from sample S1 to permit comment on the fabric of the mortar. With a thin section also prepared from mortar sample S4, a pink coloured mortar, for comparison.

A description of each sample is given in the following section of this report:



4.1 Macroscopic examination of samples

Sample SR2552-S1 – Brick sample with adhering "original" mortar

The mortar in this sample is well compacted and well bonded to the brick. Although compact the mortar could be broken down under moderate finger pressure, and once disrupted easily powdered under light finger pressure.



Plates No. 1 & 2: The above left plate shows the sample as received, with the left plate showing the mortar after being removed from the brick, and the right plate a close-up of the surface on the largest intact piece.

Water droplet tests indicated that the mortar had a well-connected pore structure with droplets placed onto freshly fractured surfaces being absorbed and diffused throughout the matrix relatively quickly. However, when placed onto masonry contact surfaces or an outer edge the water droplets were supported for a short period prior to slowly being absorbed. This may infer that either the wall had been treated at some time in the past or that water percolation across the brick/mortar interface had occurred over time with leaching and redeposition of lime within the pore structure, resulting in blockage of surface and near surface pores, which is considered the most likely cause.

On testing a freshly fractured surface of the mortar with a phenolphthalein indicator solution, the mortar was found to be fully carbonated.

The colour¹ of the mortar, was found to be 10YR 8/1 "White".

Lime inclusions are abundant, with the majority being finer than 1.0mm in size but locally inclusions up to 5.9mm were observed. The inclusions are irregular to sub-angular in shape and appear to have formed from quicklime, rather than putty lime.

The mortar displayed a high abundance of entrained air, with a low occurrence of entrapped air voids, mostly less than 3.2mm in size. The latter are indicated to be placing artefacts. None of the voids appeared to be lined with secondary minerals.

The aggregates are dominated by quartz and appear to have a maximum grain size of 1.4mm but mostly finer than 0.4mm.

¹ The colour of the mortar samples was determined against the Munsell Soil Colour Charts



Sample SR2552-S2 – Pink Mortar sample from Tower

This sample consisted of pieces of a pink coloured mortar, bonded to a piece of brick. The mortar appears to have been well compacted and adhered well to the brick surface. On removal from the brick the mortar was found to be locally very friable, with intact pieces easily powdered under light to moderate finger pressure.



Plates No. 3 & 4: The left plate shows the sample as received, which is mostly of brick with a thin layer of adhering mortar, arrowed in plate. In addition, there were small mortar fragments and powdered fines. The right plate is a close-up of an intact piece of mortar which includes lime inclusions, which range in size from 0.2mm to 3.6mm.

The mortar was found on testing to be fully carbonated and porous, with the rapid absorption of water droplets placed onto fresh surfaces. The mortar also displayed a high entrained air content, which is not uncommon in lime-based mortars.

The lime inclusions ranged from sub-angular to sub-round in shape, and as in sample S1 had the appearance of having formed from a quicklime.

The mortar colour was found to be 10R 5/3 "Weak Red".

The mortar had the appearance of a lime rich mix.



Sample SR2552-S3 – Weathered Sandstone sample

Plate No. 5:

The plate opposite is of the sandstone fragment received, which has the appearance of a fine grained pinkish red sandstone.

The sample displays a degree of weathering, which has accentuated the finely bedded structure of the stone.



The sandstone was locally soft and, where delaminating, was finger friable and it could easily be disrupted and powdered under light to moderate finger pressure. The sand grains are fine to very fine and appear to be well sorted, with the laminae highlighted by localised concentrations of mica flakes.

The sandstone was found to be 10R 5/6 "Red" in colour, on a freshly fractured surface.

Sample SR2552-S4 – Pink Bedding Mortar sample from Inside Column

This sample consisted of several intact pieces of brick bedding mortar, the mortar being well compacted, dense and firm. However, the pieces could be broken under moderate finger pressure, and, once disrupted, powdered under light finger pressure.



Plates No. 6 & 7: The left plate shows the intact pieces in the sample, as received. With the right plate showing a close-up of a freshly fractured surface through a mortar fragment. Note the abundance of lime inclusions distributed throughout the section in view.

Spot tests with a phenolphthalein solution indicated that the mortar was fully carbonated. With water droplet tests confirming a well-connected open pore structure, with their rapid absorption, and diffusion throughout the thickness of the mortar sample tested.

The mortar contains an abundance of irregular to sub-angular lime inclusions with these up to 5.2mm in size. The inclusions are similar in shape and texture to those observed in samples S1 and S2 and again appear to have formed from quicklime and locally from balled hydrate.

The mortar was air entrained with small entrapped air voids which were sparse in occurrence.

The colour of the mortar was found to be 10R 5/3 "Weak Red".

Aggregates were again dominated by sub-angular quartz grains, mostly finer than 0.5mm in size, and are visually very similar to that observed in sample S1.

Sample SR2552-S5 - Pink Mortar, from various locations within Tower

This mortar sample was visually and texturally similar to the earlier mortars, and it is likely to be from the same mix using similar components as found in samples S2 and S4.

Intact pieces ranged in size from 60mm down to <5.0mm, with the hardness varying from firm and hard, requiring persistent firm finger pressure to break, to soft and finger friable.



However, once the hard pieces were disrupted they could be powdered further under light to moderate finger pressure.



Plate No. 8:

The plate on the left shows the sample as received, which was composed of six individual containers, which we identified as S5 A to F.

Plate No. 9:

The plate opposite shows the sample received decanted from their containers, with the intact pieces separated from the powdered mortar in preparation for examination.

As with the other samples examined the mortar is fully carbonated and all the pieces tested were indicated to have a well-connected porosity from the water droplet test, with water droplets placed onto fractured surfaces being rapidly absorbed and diffused throughout. Water placed onto brick contact surfaces, were supported for a short period prior to being absorbed. The latter is considered to be due to the compacted, more dense, fabric at the brick/mortar interface.



Plate No. 10:

Close-up of a bed face on one of the intact mortar pieces, note the presence of lime inclusions distributed throughout. Inclusions are up to 3.5mm in size.

The mortar was noted to again contain an abundance of small lime inclusions distributed throughout the mortar. Inclusions ranged in size from 0.2mm to 3.5mm, with the inclusions found to range in hardness from soft and powdery to firm, but the firm inclusions could be disrupted and powdered with a point pick under moderate pressure.

The mortar is air entrained, and where observed, a low abundance of entrapped air which was sparse in abundance and random in distribution.



The colour of the mortar was assessed at 10R 6/3 "Pale Red".

Aggregates were again similar to those observed in all the previous samples examined.

Sample SR2552-S6 – Original Pink Coloured Mortar, North Side

This sample was identified as an "Original" pointing mortar and consisted of three small fragments, that were too small to break with the fingers, but once disrupted could be powdered under light finger pressure. The lime inclusions were smaller than in the other samples examined and are possibly formed from compressed and balled hydrate.



Plates No. 11 & 12: The left plate shows the sample as received, with the pencil point added for scale. The plate on the right is a magnified view of one intact piece, where the dense fabric can be seen along with an abundance of small discrete lime inclusions.

The mortar in this sample was again fully carbonated and on testing with water droplets displayed a high connected pore structure with the rapid absorption of the droplets and diffusion of the water throughout the sample tested.

In all other respects this sample was similar to that in sample S5.

The colour of the mortar was found to be 10R 6/3 "Pale Red".



Sample SR2552-S7 – Pink Repointing Mortar sample from North Side

Plates No. 13 & 14: The plate above, left, shows the sample as received, with that on the right showing a close-up of a freshly fractured surface through the thickness of the mortar, note absence of lime inclusions.



The mortar is compact and moderately hard and required moderate to firm finger pressure to break, but once disrupted could be powdered under light finger pressure, with ease. There were no lime inclusions observed in any of the fragments in this sample.

Water droplet tests confirmed a connected pore structure with very rapid absorption of droplets and their diffusion throughout the full thickness of the mortar piece tested.

A phenolphthalein indicator test showed that the mortar was fully carbonated.

The colour of the mortar was assessed at 10R 6/4 "Pale Red".

The aggregates were again quartz dominated, with minor proportions of dark minerals present. The grains are slightly coarser than those in the earlier samples examined, with a higher proportion in the 1.0 to 2.2mm fraction, with the grains being sub-round in shape with abraded water worn surfaces.

Sample SR2552-S8 – Pink Mortar sample above Sandstone Masonry

This sample was compact and firm which on breaking was found to break with an audible 'snap' but once broken could be powdered with ease. The mortar contained a few small lime inclusions, with the largest being 3.2mm in size.



Plates No. 15 & 16: The left plate is a view of the sample as received, with the right plate showing a magnified view of freshly fractured surfaces through the thickness of the sample. The lower piece had been tested with a phenolphthalein indicator to test for carbonation.

The mortar was found to be fully carbonated and on the basis of a water droplet test was indicated to have a well-connected open porosity.

The mortar colour was found to be 10R 6/4 "Pale Red" in colour.

Aggregates were fine grained and again dominated by quartz, and were similar to those in samples S1 to S6.



5.0 Microscopic Examination

Petrographic thin sections were prepared from slices sawn from two pieces of mortar, with the slices aligned to permit the maximum area of the mortar, on the slides.

The samples were prepared for thin sectioning by initially drying them to a constant weight at 60°C prior to impregnating the dried sub-samples with an epoxy resin containing a fluorescent blue dye.

One side the impregnated slices were polished and mounted onto glass slides (50mm x 75mm). The mounted samples were then ground and polished to a thickness of approximately 30microns.

The microscopic examination of the thin sections was undertaken using an Olympus BH2 Polarised light microscope, fitted with a Digital Camera, to permit the recording of images of areas of note for record purposes.

The presence of dyed epoxy resin enables detailed analysis of void distribution, an assessment of microporosity and a clear indication of any crack patterns present, in plane polarised light.

The samples were also mounted onto a 'Swift automatic stage' which permitted the determination of the volumetric mix composition, by modal analysis.

The observations from the examination of the samples are presented below:

5.1 Sample SR2552-S1 – Grey Mortar Adhering to Brick Sample

This sample was composed of an intact section of mortar adhering to a clay brick, with the section prepared from mortar separated from the bed face of the brick.



Plate No. 17:

Thin section prepared from a piece of mortar from Sample S1 for microscopic examination.

Aggregate

The aggregate is composed of a mixed suite of minerals, dominated by quartz, with feldspar, including Plagioclase, Microcline and Orthoclase along with heavily altered volcanic igneous rock fragments, granite, and microcrystalline silica (chert). There is also a proportion of weathered sandstone (greywacke) and indeterminate weathered metamorphic grains. The grains are sub to rounded to rounded in shape, with smooth water worn surfaces.

A very low proportion of chlorite and kaolinite clay minerals were also observed, but in trace proportions, incorporated within heavily altered minerals.



A trace proportion of heavy minerals were also indicated to be present and these are typical of Zoisite/Epidote.

The sand is a quartz rich natural sand with a proportion of lithic fragments, including amphibole.

Binder

The binder is of a non-hydraulic lime, with the binder containing an abundance of incompletely burnt limestone fragments. There is also a low abundance of incompletely slaked lime inclusions. The inclusions are sub-angular to sub-round in shape and their appearance and texture would suggest that the quicklime had been slaked with the sand, and not prepared as a hydrate or run to a putty before use. The mortar has <u>not</u>, however, been placed as a Hot Mixed Mortar (HMM), but mixed as such, with the lime slaked with the sand, banked to cool and fully hydrate the quicklime, as much as practical, using this technique. With the sand lime mixture probably rescreened, to remove oversize and unslaked lime, prior to being mixed and retempered, with the mortar placed cold. This practice was commonly used by bricklayers in the 18th and 19th centuries.

Although the paste in the binder is fully carbonated, some of the incompletely slaked, or incompletely calcined lime inclusions appear to retain cores of incompletely carbonated lime. A low proportion the lime inclusions were also noted to contain fine clusters of dolomitic rhomb's, which may infer that the limestone was impure, and feebly dolomitic.

There is no evidence of alteration products within the body of the mortar, albeit there is evidence of partial dissolution and the redeposition of calcite within a few of the smaller voids. Within these fine acicular crystals were observed, which may infer that minor reaction products, from a reaction of the binder and acid rain or other environmental sulphates, had occurred. This though is very minimal in the sample examined, and perhaps below the detection level of the other analytical procedures used in the mortar analysis.

Voids and microcracks

Voids are abundant and are present, both as large interconnected spaces (up to 4.7mm in size) and as small discrete entrained air bubbles (typically <0.1mm in size) entrapped within the paste in dense patches of matrix.

Cracks are rare, and where present are randomly distributed throughout; they range in width from <0.02mm to 0.16mm and have the appearance of drying shrinkage cracks.

Cracks and most voids are free of secondary minerals and there was no evidence of significant fluid migration through the mortar sample.

The results of a point count (modal) analysis carried out on the thin section are presented in the following table:



Sample Ref:	SR2552-S1			
Constituents		%		
Aggregate	Inclusions as binder	Inclusions as aggregate		
Quartz	52.6	52.6		
Feldspar	4.1	4.1		
Igneous rock fragments	8.4	8.4		
Opaque Minerals	0.8	0.8		
Lime Inclusions & Clinker	- 5.9			
Total Aggregate	65.9	71.8		
Binder (Lime Paste)	28.2	28.2		
Clinker	0	0		
Lime Inclusions	5.9	0		
Secondary products/Calcite				
sulphate, etc	0	0		
Total Binder	34.1	28.2		
Total Constituents	100.0	100.0		
Voids	16.8	16.8		
Crack	1.8	1.8		
Cracks/Voids	18.6	18.6		
Mix	Composition, by volum	e		
	Total Binder	Effective Binder		
Binder: Aggregate Ratio	1.0 : 1.93	1.0:2.55		

Table No. 1: Modal Analysis carried out on thin section prepared from sample S1.

Photomicrographs:



Plate No. 18:

A view in plane polarised light (ppl), of a typical area of the mortar where the highly voided condition of the mortar can be seen, highlighted by pigmented impregnating resin.

Most of the sand grains in view are quartz (white in section), with volcanic igneous grains and feldspar also apparent. A large partially calcined lime inclusion is seen in the lower left of the plate.

Note the low apparent paste content, although all grains are partially coated.

Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.









Plate No. 19:

Another view in ppl, of an area of paste containing binder showing a clotted appearance where compressed between sand grains. The aggregates in view are again dominated by quartz, with feldspars and other lithic particles.

A small partially slaked lime inclusion can be seen in the upper centre of the plate, which on examination under high magnification was noted to contain Portlandite, uncarbonated lime. Porosity is highlighted by the blue dyed resin. Field of view 1.2mm.

Plate No. 20:

A view in cross polarised light (xpl) of an area of the mortar where the paste is abundant and in this view is fully carbonated.

Aggregate grains are again dominated by quartz, with altered igneous fragments along with a partially calcined limestone fragment, upper right.

The bright coloured mineral in centre right is Zoisite.

Voids and the blue dyed resin appear dark in cross polarised light. Field of view 1.2mm.

Plate No. 21:

Another view in cross polarised light (xpl) of a more open textured area towards the outer surface of the mortar. The paste is less dense and again fully carbonated. Quartz grains are also apparent, along with feldspar and altered volcanic igneous grains. A fully hydrated lime inclusion can be seen in the upper centre, at the margin, with a small intact inclusion also apparent lower left of centre. Voids and the blue dyed resin appear dark in cross polarised light.

Field of view 1.2mm.



5.2 Sample SR2552-S4 – Sample of Pink Coloured Mortar



Plate No. 22:

Thin section prepared from a piece of mortar from Sample S4 for microscopic examination and composition by modal analysis.

This sample was well compacted and moderately hard, resisting breakage under light finger pressure. The mortar was noted to contain an abundance of lime inclusions, which from their appearance, would infer that the mortar had been made using a quicklime as the binder.

Aggregate

The aggregates in the mortar sample consist of a mixed suite of rock types, as in sample S1, and are again dominated by quartz, with volcanic igneous rock fragments, feldspar, granite, chert and minor amphibole, with trace proportions of muscovite mica and heavy minerals such as Zoisite/Epidote. A proportion of clinker was also observed, but given its occurrence and association with lime inclusions it is considered that this was included with the lime rather than the aggregate. Some of the heavily altered igneous and metamorphic grains have weathered to chlorite and other clay minerals, but these minerals are still mostly encapsulated within the altered sand grains.

The aggregates are sub-angular to sub-round in shape, with a low proportion of elongated aggregate particles also present. The shape and texture of the aggregates suggesting a fluvial deposit as the source for the sand. Aggregates range in size from 0.06mm to 1.2mm (coarse silt to coarse sand). As there was minimal clay and fine silt materials observed, it is again inferred that a water transported material was used as the aggregate. The aggregates are mostly well bound within the paste.

The mortar contains a high proportion of dark/opaque minerals, which range in size from 0.01mm to 0.1mm in size, with the shape ranging from angular and flaky to irregular and these are considered to be Hematite flour added to the mix as a pigment.

Binder

The binder is typical of a lime mortar, with an abundance of lime inclusions apparent within the section. A significant number of the inclusions appear to have formed from non-hydraulic quicklime, however, there is a proportion of the inclusions that contain hydraulic components, with some of the incompletely slaked inclusions observed to retain a faint imprint of the rock fabric, suggesting that a proportion of a clayey limestone was incorporated within the feed stock. There was no evidence of dolomitic limestone.

Several the inclusions appear to have been over burned with these also associated with clinker in the paste, which may have originated from the fuel used in the kiln.

The paste is fully carbonated and unhydrated lime inclusions were not observed.



The lime inclusions apparent, include both sub-round and sub-angular inclusions, with most having the appearance of having formed from incompletely slaked quicklime, with others fully hydrating and appearing as unmixed putty inclusions.

The more angular inclusions, however, are typical of particles of incompletely slaked quicklime, at the time of placing. Lime inclusions range in size from 0.2mm to 3.6mm in the section examined, with most of these showing well defined margins with little evidence of diffusion into the surrounding paste, but without displaying perimeter cracking. Whereas, most of the rounded fully slaked quicklime inclusions (putty) display partial diffusion into the surrounding paste and/or peripheral (shrinkage) cracks.

From the examination of this sample it is indicated that the binder was probably mixed with the aggregate, this in the form of a quicklime, but it was well slaked, cooled and probably screened prior to being remixed in preparation for placing.

Voids and microcracks

Voids are abundant as both entrapped air voids, which are typically round to sub-round in shape and range from 0.08mm to 2.4mm in size, but mostly <1.2mm. Some localised rounded voids were observed to be lined by fine fringes of calcite, with very localised clusters of gypsum crystals.

Cracks are rare and occur as localised features peripheral to incompletely slaked quicklime particles. The cracks are fine, ranging in width from <0.03mm to 0.10mm, and are typical of drying shrinkage features.

Sample Ref:	SR2552-S4			
Constituents		%		
Aggregate	Inclusions as binder Inclusions as Aggre			
Quartz & Flint	54.5	54.5		
Feldspar	2.8	2.8		
Igneous rock fragments	7.6	7.6		
Opaque Minerals	2.0	2.0		
Dolomitic Limestone	-	0		
Lime Inclusions & Clinker	0	7.1		
Total Aggregate	66.9	74.0		
Binder (Lime)	26.0	26.0		
Clinker	2.7	0		
Lime Inclusions	4.4	0		
Secondary products/Calcite				
sulphate, etc	0	0		
Total Binder	33.1	26.0		
Total Constituents	100.0	100.0		
Voids	14.7	14.7		
Crack	0.6	0.6		
Cracks/Voids	15.3	15.3		
	Total	Effective		
Binder: Aggregate Ratio	1.0 : 2.02	1.0:2.84		

The results of a point count (modal) analysis are presented in the following table:

Table No. 2: Modal Analysis carried out on thin section prepared from sample S4.



The effective binder content is calculated on the basis that the inclusions are acting as aggregate rather than as binder and is probably a truer measure of the binder content of the mix, relating to its performance as a mortar. Whereas, the total lime content reflects the mix at the time the mortar was made and placed, including the inclusions (both fully slaked and unslaked) as part of the added lime binder.

Photomicrographs:





Plate No. 23:

A view in plane polarised light (ppl) showing a typical area of the mortar, with a partially slaked angular lime inclusion, in the centre of the plate, which contains Belite clusters, confirming a hydraulic component in the binder. The aggregates are dominated by quartz in this view, with minor feldspar and igneous fragments.

A high proportion of fine opaque grains are noted distributed throughout the paste, with this the Hematite added as a pigment.

Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

Plate No. 24:

A magnified view of a binder rich area of the mortar. Here the paste appears darker due to the high pigment content.

Belite clusters can be seen clearly within the paste, arrowed in plate, confirming that the mortar was made from a hydraulic binder. In addition to this there is a high concentration of Hematite within the paste, making it appear very dark. The aggregates in view are mostly of quartz.

A partially infilled void can be seen in the centre of the plate, with this containing secondary minerals, gypsum.

Porosity is highlighted by the blue dyed resin. Field of view 1.2mm









Plate No. 25:

A view in Cross polarised light (xpl), of an area containing a clinker grain, centre of plate, which can be seen to contain quartz grains and other indeterminate minerals. The clinker is surrounded by a fringe of calcite, which also permeates porous zones in the clinker.

An unmixed fully slaked lime inclusion is apparent in the upper right.

Aggregates are dominated by quartz, with feldspar and altered igneous fragments.

Voids and the blue dyed resin appear dark in cross polarised light. Field of view 1.2mm.

Plate No. 26:

A view in Plane polarised light (ppl), of an area containing two lime inclusions, the upper centre inclusion contains Belite and other clinkered components, whereas that in the lower centre is composed of a nonhydraulic lime.

Belite clusters are also observed within the paste compressed between aggregate grains.

Aggregates are dominated by quartz, with feldspar and altered igneous fragments also present.

Porosity is highlighted by the blue dyed resin. Field of view 1.2mm.

Plate No. 27:

A magnified view in Plane polarised light (ppl), of an area within the edge of the upper lime inclusion in plate No. 26 above.

Here the presence of partially hydrated Belite (C2S) can be seen within the darker paste, arrowed in plate.

The paste to the left is fully carbonated and can be seen to be separated from the inclusion by a fine shrinkage crack

Porosity is highlighted by the blue dyed resin. Field of view 0.4mm.



6.0 Analysis by X-Ray Diffraction

To assist in the identification of the composition of the binder in the mortar samples, a binder rich sub-sample from each sample was prepared, from a typical example of each variant received, and these submitted to analysis by X-ray Powder Diffraction (XRD).

To achieve this, a representative sub-sample of the mortar was obtained, from each sample, with these ground in an agate mortar and pestle, taking care to minimise crushing of the aggregate. A concentrated binder rich sub-sample was obtained by sieving the sample over a 63µm sieve. The powder collected was then back-packed into proprietary sample holders for presentation in the X-Ray Diffractometer. This technique being used to ensure the random orientation required to give true peak intensities in the diffractograms.

In addition, a sample of the sandstone was also prepared and analysed by XRD to confirm the form of the matrix minerals present.

A sample of the fines recovered, following acid digestion and grading, from one of the pigmented mortar samples, was also analysed. This was to clarify the composition of the fines in the sample, minus the binder, to permit identification and quantification of the pigment used in the pigmented mortar samples.

The prepared samples were analysed in a Philips X-ray Diffractometer fitted with a single crystal monochromator, set to run over the range 3° to 55° 2θ in steps of 0.1° 2θ at a rate of 1° 2θ /minute using CuK α radiation. With the digital output from the diffractometer analysed in a computer program, which matched the peak positions against the JCPDS International Standard Mineral Data-base sub-files using a search window of 0.1° .

The result obtained are shown in the following figures appended to the end of this report, in the form of a labelled X-ray diffractograms:

Figure No. 4 – SR2552-S1 – "Original" Grey bedding mortar, adhering to brick from Attic,
Figure No. 5 – SR2552-S2 – Small pieces of "Pink" coloured mortar, from inside Tower,
Figure No. 6 – SR2552-S3 – Piece of sandstone spalled from foundation stone masonry,
Figure No. 7 – SR2552-S4 – "Pink" Brick bedding mortar from inside column, on Tower,
Figure No. 8 – SR2552-S5 – "Pink" Bedding & Pointing mortar various locations in Tower,
Figure No. 9 – SR2552-S6 – "Original Pink" mortar from Brick masonry North side,
Figure No. 10 – SR2552-S7 – Repointing "Pink" coloured mortar from North side of Structure,
Figure No. 11 – SR2552-S8 – "Pink" mortar sample from Stone masonry, West elevation,
Figure No. 12 – SR2552-S5A – Fines passing 63µm sieve, from sample S5 after acid digestion.

The most abundant mineral components identified are indicated in the appended diffractograms using the following short-hand notation. With the exception that in sample S5 there was insufficient data space to label all peaks and, therefore, three data base lines are included to highlight the position of those peaks not independently labelled.

- cc = Calcite (CaCO₃) Calcium Carbonate, carbonated lime binder component, also the dominant component of any limestone in the aggregate,
- ar = Aragonite (CaCO₃) another crystalline form of Calcium Carbonate, commonly associated with shell, but also formed from leached and redeposited carbonated lime,



- **po** = Portlandite (Ca(OH)₂) Calcium Hydroxide, hydrated uncarbonated lime from binder,
- $\mathbf{br} = \text{Brucite } (\text{Mg}(\text{OH})_2) \text{ Magnesium Hydroxide, hydration product in a dolomitic lime,}$
- $be = Belite (Ca_2SiO_4) di$ -calcium Silicate, Clinker component in Portland cement and some Hydraulic lime binders,
- fr = Friedel's Salt (Ca₄Al₂O₆Cl₂H₂O) Calcium Aluminium Oxide Chloride Hydrate, ion exchange product in aluminous binders and altered volcanic minerals in contact with chlorides,
- $zo = Zoisite (Ca_2Al_3(SiO_4)(Si_2O_7)(O,OH)_2)$ Calcium Aluminium Silicate Hydroxide, Common epidote mineral in metamorphic rocks, and also rarely in slags and ashes And, if from the latter, can contribute to hydraulicity in some mortars,
- **gy** = Gypsum (CaSO₄2H₂O) Calcium Sulphate Hydrate, most likely present as reaction product between lime in binder and environmental sulphates or sulphates leached from the brick,
- do = Dolomite (Ca,Mg(CO₃)₂) Calcium Magnesium Carbonate, dominant component of Dolomitic limestone and in carbonated Dolomitic lime binder,
- $he = Hematite (Fe_2O_3)$ Iron Oxide, dominant component of the Red/Pink pigment used to colour the mortar, with trace proportion also found in the pink sandstone,
- **fs** = Feldspar, mostly of the Alkali Feldspar group, Sanidine, Orthoclase, Microcline are common in all samples, with minor Plagioclase group minerals Anorthite and Albite also present, all common rock forming mineral, present as components of the aggregate,
- **ka** = Kaolinite, Clay mineral perhaps present as an aggregate component, or from soiling,
- **di** = Dickite, Clay mineral of the Kaolinite group of minerals, aggregate component, or present as soiling,
- **mi** = Muscovite mica, Common rock forming mineral, found in granites, etc.
- $si = Siderite (FeCO_3)$ Iron Carbonate, aggregate component, added with the pigment or a reaction product from binder acting on pigment,
- $an = Ankerite (Ca(Fe,Mg)(CO_3)_2)$ Calcium Iron Magnesium Carbonate, Ankerite forms with Siderite in low grade metamorphosed iron rich sediments,
- $fa = Fayalite (Fe_2SiO_4)$ Iron Silicate, mineral of the Olivine group, perhaps present as an aggregate component or as a contaminant in the pigment added to the mortar,
- qz = Quartz (SiO₂) dominant component of the sand grains in the aggregates used in all of the mortar samples, and dominant mineral present in the sandstone sample.

From the analysis it is indicated that there are a range of binder variants present in the samples submitted for analysed.

Sample SR2552-S1: The result of the analysis indicates that the unpigmented "Grey/White" mortar in sample S1, bedding mortar, is a non-hydraulic, feebly dolomitic, lime, which appears to have been maintained in a damp condition, for an extended period, as there is still uncarbonated binder components present, in the form of Portlandite and Brucite. When fully carbonated the portlandite converts to Calcite, and the Brucite converting to Dolomite. The binder appears to be non-hydraulic.

Samples SR2552-S2, S4 and S5 all appear to contain a binder that is feebly to moderately hydraulic, with the presence of Belite (C_2S), a clinker component in some hydraulic limes and Portland cements detected. However, in the absence of Alite (C_3S) and other common hydration products, it is indicated that the binders are most likely hydraulic lime.



The absence of the most common Aluminates in these samples would also discount a Natural cement as the binder, or a gauging in a lime binder. The binder in all three samples are fully carbonated and the binders were also found not to be dolomitic, suggesting a different lime source to that used in sample S1.

The aggregates in samples S2 and S4 are similar in mineral composition to those in sample S1, and this may suggest that these mortars were perhaps from the same period of construction. All contain Quartz, Friedel's salt and the Feldspars are dominated by Sanidine with minor Plagioclase and Orthoclase also present. Whereas, the aggregates in sample S5 differ in that in addition they contain, a low proportion of Dolomite and Kaolinite along with the Feldspar minerals Anorthite and Albite, which may infer either a different aggregate source, or a different horizon within the same source, or perhaps indicate that this mortar was from a different construction period, though the latter is discounted by the similarity of the binder.

Samples SR2552-S4 and S5 also both contain Gypsum, which is probably present as a reaction product formed from the reaction of lime (from the binder) with either environmental sulphates (Smoke pollution or acid rain) or sulphate leached from the brick. The presence of a trace proportion of Siderite in sample S5, may be present as a contaminant or a reaction product, or a component of the aggregate, but this was not detected in any of the other samples.

Sample SR2552-S6 is from a non-Hydraulic, non-Dolomitic, lime mortar. This sample was identified as an "Original" mortar, which would perhaps place it at the same construction period as sample S1, albeit the binder used at this location was not dolomitic. The presence of Fayalite in the aggregate, which was not found in any of the other samples analysed, may again infer that the aggregates came from a different source, or horizon, as this mineral is commonly found in volcanic rocks and metamorphosed iron rich sediments. If the Fayalite had been incorporated with the Hematite pigment, the aggregates used in this sample would otherwise be similar to those in S1, confirming the hypothesis that it was an original mortar placed at the same time as sample S1.

Sample S7 does not contain Belite (C_2S) and is indicated to have been made using a non to very feebly hydraulic lime as the binder. This sample was also found to contain Aragonite, which may additionally infer the presence of shell in the aggregate, or that limestone containing shell was used in the production of the lime, and a proportion was not completely calcined, but this was not detected in any of the earlier samples analysed. Therefore, there is also the possibility that it had formed due to leaching and redeposition of the carbonate within the pore structure of the mortar, or deposited on contact or crack surfaces.

Although there was insufficient sample to make a thin section to confirm binder form used, it was indicated, from the examination of the sample under the stereo-microscope, that this mortar was mixed using a binder in the form of a hydrate.

Sample S8 was again a non-hydraulic lime mortar, however, it was found to contain a trace proportion of Dolomite, similar to that in sample S1, but at very low concentration (0.9% of the fines dominated analysis sub-sample), and therefore this may be from an aggregate component or be present as a contaminant, rather than the binder.



There is also the possibility that, as in sample S1, that the limestone burned to produce the lime was a 'dirty' limestone containing trace proportions of Dolomitic minerals, along with, or within the clays observed in some of the lime inclusions.

Sample S8 again was found to contain aragonite, but this is considered to be most likely present as a redeposited leached binder product, rather than an aggregate component.

The Pigment found in all "Pink" mortar samples is Hematite, and this is not an uncommon pigment used for this purpose. The source of the pigment is not considered to be the Pink sandstone used in the foundations, as, although, this also contains hematite, the concentration is very low, see Figure No. 6 at the end of this report. The very fine grain size of the sandstone along with the other mineral components present in the stone, not found in the mortar, would again tend to exclude this as the source of the pigmenting additive included in the mortars.

The results of the analysis of the fines (passing the 63-micron sieve) recovered after acid digestion of sample S5, again confirmed that the pigment used was Hematite, see Figure No. 12. The absence of Muscovite, Dickite, Ankerite and Gibbsite would, however, again confirm that fines from the sandstone had not been added to the mortar as a pigment.

The data from the XRD analysis was further processed by Rietveld Refinement. This permitted quantification of the components present and was used to determine the proportion of pigment in the samples analysed. By correcting the quantity of the pigment found in the fines, for the mean proportion of fines, determined from the acid digestion, the following values are indicated to be the proportion of Hematite pigment added to each sample analysed:

Sample Ref.	S2	S4	S 5	S6	S7	S8	Mean Value
Hematite (% by mass)	0.29	0.35	0.31	0.30	0.16	0.31	0.26

7.0 Mix Composition Analysis

A total of three samples were prepared for mix composition by acid digestion, these were samples SR2552 - S1, S4 and S5, i.e. those having sufficient material to permit acid digestion, with recovery of aggregate, for grading analysis.

A further four samples were submitted to analysis by the methods of BS4551, however, the results obtained should be used with caution, and for comparative purposes only, as the results of the analysis are calculated on the basis of data pertaining to modern Hydraulic limes, as there was insufficient inclusions present, of sufficient size, to permit their extraction for independent analysis.

The results of the analysis carried out are presented in the following sections:



7.1 Mix Composition Analysis by Acid Digestion

Sample Ref.	SR2552-S1	SR2552-S4	SR2552-S5	
	Bedding	Bedding	Pointing	
Binder Type	Non-Hydraulic	Moderately	y Hydraulic	
Lime : Aggregate Ratio	1.0:3.9	1.0:4.1	1.0:3.7	
Weight Proportions – Qui	cklime Mixes			
Binder content	1.0	1.0	1.0	
Fine Aggregate (Sand)	7.1	6.1	5.5	
Volume Proportions				
Binder content	1.0	1.0	1.0	
Fine Aggregate (Sand)	2.2	1.9	1.8	
Weight Proportions – Lim	e Hydrate Mixes			
Binder content	1.0	1.0	1.0	
Fine Aggregate (Sand)	5.0	4.9	4.5	
Volume Proportions				
Binder content	1.0	1.0	1.0	
Fine Aggregate (Sand)	2.0	2.2	2.0	

The mix proportions determined by this method includes all lime inclusions and any readily acid soluble aggregate as binder. Therefore, the results obtained tend to be lime rich, particularly where lime inclusions are present, and the binder content should be reduced by a factor to correct for this.

The result of the grading analysis carried out on the aggregate recovered following acid digestion, are presented in the table below and as aggregate filled histograms in the appended Figures No. 1, 2 & 3.

Sample Ref:	SR2552-S1		SR2552	2-S4	SR2552-S5		
BS Sieve Size (mm)	Retained %	Passing %	Retained %	Passing %	Retained %	Passing %	
8.00	0	100	0	100	0	100	
4.00	0.5	99.5	0	100	0	100	
2.00	0.9	98.6	0.6	99.4	0.2	99.8	
1.00	7.8	90.8	0.9	98.5	1.3	98.5	
0.500	7.1	83.7	7.4	91.1	7.6	90.9	
0.250	40.3	43.4	43.4	47.7	43.7	47.2	
0.125	37.8	5.6	39.7	8.0	39.7	7.5	
0.063	2.9	2.7	3.4	4.6	3.4	4.1	
Passing 63µn	n 2.7		4.6		4.1		

Based on the particle size distribution it could be inferred that as the aggregate gradings are very similar, and that they are likely to be from the same, or a very similar source, albeit that there is a minor variation in the minerals present. All have relatively low fines content, bearing in mind that the proportion passing the 63-micron sieve will also contain the pigment added to the mortars. This may, therefore, suggest, that due to the low clay and silt content, the sand was either from a processed source, i.e. screened and washed, or more likely, given the age of construction, infer that the sand had been extracted from water transported deposit.



7.2 Mix Composition by chemical Analysis

As there was insufficient sample for acid digestion in all samples, small sub-samples from samples S7 and S8 were submitted to analysis by the methods of BS 4551:2005 + A1:2010 & A2: 2013. In addition, a further sub-sample from samples S4 and S5 were also analysed by this method for comparison with the data obtained by the other methods employed. The results obtained are reproduced below:

Sample Ref.	SR2552-S4	SR2552-S5	SR2552-S7	SR252-S8		
Chemical Analysis	% by mass					
Insoluble Residue	79.88	79.12	76.83	74.99		
Soluble Silica (SiO2)	0.91	1.04	2.04	1.90		
Calcium Oxide (CaO)	5.45	5.65	8.68	10.30		
Loss on Ignition	8.88	9.30	9.56	10.23		
Approximate volume Propo	ortions calculate	d on the basis o	f standard assur	nptions.		

Hydraulic Lime	1.0	1.0	1.0	1.0
Sand	3.8	3.6	2.0	1.7

Comments

The analytical results presented above were evaluated by the method of BS 4551: 2005 + A1: 2010, on the basis of the following assumptions, with the values reported calculated from the soluble silica content:

- a. The Hydraulic Lime content, for samples S4 and S5 has been calculated on the basis that a Moderately Hydraulic binder contained 10.5% soluble silica and 65.1% calcium oxide and had a dry bulk density of 670 kg/m³, with samples S7 and S8 calculated on the basis of a Feebly Hydraulic lime containing 7.6% soluble silica, 66.6% calcium oxide and a bulk density of 580kg/m³.
- b. It was assumed that the sand contained 0.2% soluble silica and no soluble calcium compounds and had a dry bulk density of 1675kg/m3.
- c. The mortar contained no mineral admixtures.

However, the results presented above must be treated with caution as there was insufficient sample to allow recovery of the lime binder for chemical analysis. Therefore, the chemical composition of modern Feebly Hydraulic (NHL2) and Moderately Hydraulic Limes (NHL3.5), in the form of hydrates, have been used in these calculations.

8.0 Summary

The mortars used in the samples appear to have been made from a mixture of binders and this may reflect the time that the works were executed, with these including High Calcium and a feebly Dolomitic limes, ranging from non to feebly, and moderately hydraulic.

It is considered that some of the mortars (S1, S6 and S8) may have been mixed using nonhydraulic quicklime, with samples S2, S4 and S5 made with a hydraulic lime. Sample S7 has been mixed from a lime hydrate, with the binder being a non-to feebly hydraulic binder.



Samples S1 and S8 although non-hydraulic appeared to be feebly dolomitic, with the binder containing approximately 3% Brucite + Dolomite in sample S1 and 0.9% in sample S8, which is well below the values expected in a Dolomitic lime produced from a Dolomitic Limestone or Magnesian Limestone.

Details of the mixes are summarized below:

Sample Ref.	S1	S2	S4	S 5	S6	S7	S8
Volume Proportions (by acid d	igestior	ı — Tot	al Binder	conten	t – Quic	klime B	inder)
Binder content (Quicklime)	1.0		1.0	1.0			
Fine Aggregate (Sand)	2.2		2.2	2.0			
Volume Proportions (by Chemi	Volume Proportions (by Chemical Analysis – Total Binder content – NHL Hydrate)						
Binder content (Quicklime)			1.0	1.0		1.0	1.0
Fine Aggregate (Sand)			3.8	3.6		2.0	1.7
By modal analysis							
Binder: Agg. by vol. (Total)	1.0:1.9	9	1.0:2.0				
(Effective)	1.0:2.0	6	1.0:2.8				

Form of Binder

Samples S1, S6 & S8 - Non to Feebly Hydraulic Calcium lime used as quicklime, with the lime possibly being feebly dolomitic.

Samples S2, S4 and S5 – Moderately Hydraulic Calcium Lime, used as a quicklime, with no Dolomitic component.

Sample S7 – non to Feebly Hydraulic lime, not dolomitic, used as a hydrate.

On the basis of the analysis carried out it can be confirmed that none of the samples received, and analysed, were made from, or contained, Portland cement or Natural Cements as the binder.

Aggregates

All the aggregates in the masonry mortars are quartz rich natural aggregates, and although there are minor differences in particle size in samples S1, S4 and S5 they consist of a mixed suite of rocks, with minor mineral variation, and these may have been obtained from the same local source.

Visually the grain sizes in the other samples, with the exception of sample S7, all appear visually similar to the earlier samples, with respect to grain size and shape albeit there are differences in their mineral composition. This though may not be sufficient to infer a different construction period or aggregate source.

Knowledge of the local geology would be required to assess this further, to identify the source and determine if the mineral variations are such as to infer different sources, or periods of construction.

However, the aggregate in sample S7 appears to be a processed aggregate, composed essentially of quartz, with minor proportion of other minerals present, this may be from a different source, or at least, a processed material from the same source.

Pigment

The pigment used to colour samples S2 and S4 to S8 is Hematite with the form used being coarsely ground and not as fine as that used in modern pigments, see Petrographic report on sample S4 in section 5.2. Although the sandstone sample provided also contains Hematite, it is at a low concentration and was not that used to pigment the mortar used in the works.



9.0 Quality Statement

We confirm that in the preparation of this report we have exercised reasonable skill and care.

The observations, analysis results, and comments offered herein, relate only to the samples of mortar received from Berggren Architects, which were identified as having been obtained from the masonry mortar in the Wayne County Courthouse, Nebraska and were received in CMC's Stirling Laboratory on the 1st February 2018.





Figure No. 1: Grading of Aggregate from sample SR2552-S1





Figure No. 2: Grading of Aggregate from sample SR2552-S4





Figure No. 3: Grading of Aggregate from sample SR2552-S5





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Figure No. 4 – SR2552-S1 – "Original" Grey bedding mortar, adhering to brick from Attic.





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Figure No. 5 – SR2552-S2 - Small pieces of "Pink" coloured mortar, from inside Tower.

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Figure No. 6 – SR2552-S3 – Piece of sandstone spalled from foundation stone masonry.





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Figure No. 7 – SR2552-S4 – "Pink" Brick bedding mortar from inside column, on Tower.

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Figure No. 8 – SR2552-S5 – "Pink" Bedding & Pointing mortar various locations in Tower.





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Figure No. 9 – SR2552-S6 – "Original Pink" mortar from Brick masonry North side.

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Figure No. 10 – SR2552-S7 – Repointing "Pink" coloured mortar, from North side of Structure.

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Figure No. 11 – SR2552-S8 – "Pink" mortar sample from Stone masonry, West elevation.

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Figure No. 12 – SR2552-S5A – Fines passing 63µm sieve, from sample S5 after acid digestion.

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APPENDIX "A"

Sample Locations:

Plates Showing Sample Locations

Image No	Sample reference
2241 & 2685	SR2552 – S4
2212 & 2211	SR2552 – S5
2652	SR2552 – S6
2147 & 2651	SR2552 - S7
2650	SR2552 – S8





Image 2241 – Sample S4



Image 2685 – Sample S4





Image 2212 – Sample S5



Image 2211 – Sample S5

Image 2652 - Sample S6

Image 2147 – Sample S7

Image 2651 – Sample S7

Image 2650 – Sample S8