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Our Ref: M/1571/14/C1

15<sup>th</sup> March 2015

## CERTIFICATE OF ANALYSIS AND EXAMINATION OF MASONRY SAND

Project Reference	:	York House
Sample Source	:	Plaster on Hard, from Stair
Sample Description	:	Clay/lime Plaster
Date Received	:	17 <sup>th</sup> December 2014
Sample Ref	:	SR2211 – S1
Date of Test	:	Various dates, 17 <sup>th</sup> December 2014 to March 2015.
Method of Test	:	Determination of mix composition by acid digestion, with further confirmation by modal analysis on a Petrographic thin section. XRD Analysis by an In-house procedure.

### Samples

A batch of Clay/Lime and Clay mortars and plaster was received from Nigel Copsey of the Earth, Stone & Lime Company on the 17<sup>th</sup> December 2014. The samples were provided to assist with an assessment of the composition of clay and clay/lime mortars. These were requested to permit the mortars to be examined for comparison with mortars from various other locations in the UK. This report presents the results of the examination and analysis of one sample of clay/lime plaster sampled from York House.

On receipt in the laboratory, the sample details were entered into the sample register and the unique sample identification number SR2211 allocated. It was stated that the sample was from a clay/lime plaster, on the Hard, from the Stair at York House.

### Method of Test

On receipt the sample was examined with the aid of a stereo-binocular microscope at magnifications up to x20. During the examination the samples response to a selection of reagents and indicator solutions were noted.

Following the initial examination a sample of the binder was prepared for analysis by X-Ray Diffraction (XRD) to establish the mineral composition of the binder. A further sub-sample of the mortar was also obtained and this digested in dilute acid and the insoluble residue recovered. This was to arrive at a mix composition for the plaster and permit grading of the recovered aggregate/clay component in the plaster.

A petrographic thin section was also prepared from a representative slice cut from an intact piece of the plaster for examination in the polarised light microscope.

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### Observations from Macro/Microscopic examination



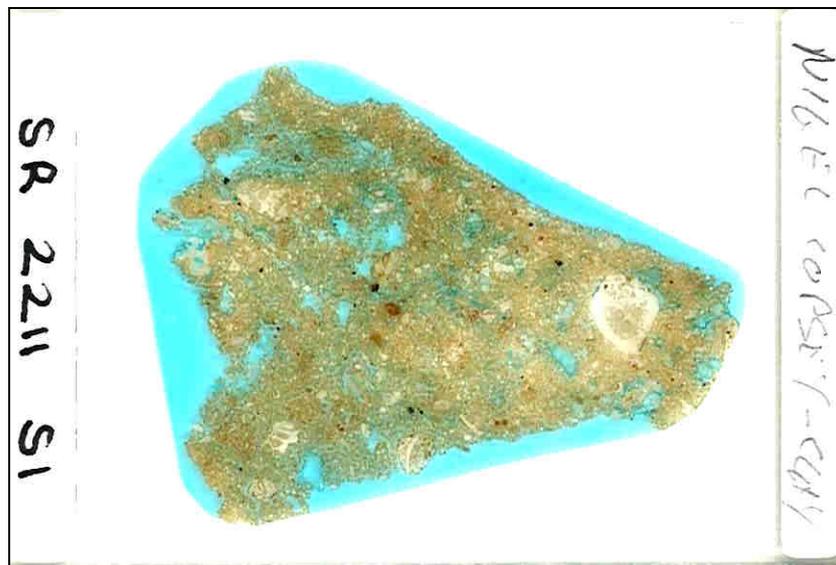
**Plate No. 1:** The above shows the outer surface of the sample as received, prior to drying. Note the high abundance of lime inclusions, the inclusions mostly have rounded margins and from the initial examination it is indicated that the lime was probably used in the form of a putty lime, width = 64mm.



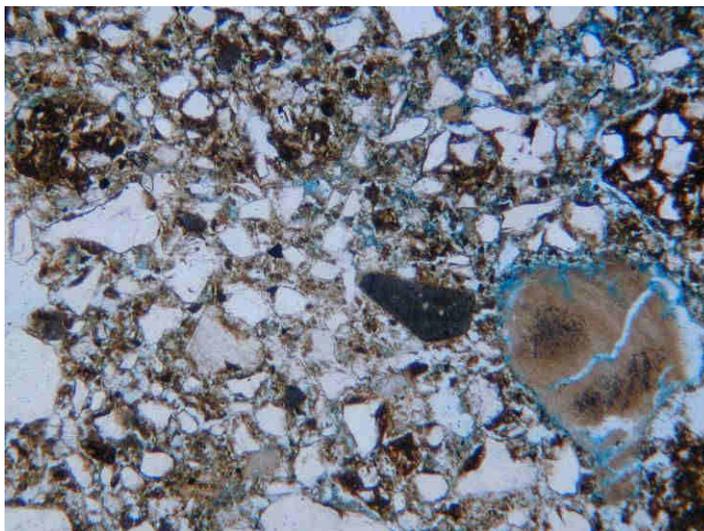
**Plate No. 2:** This shows the sawn surface through the plaster, from which a slice was removed for the preparation of a petrographic thin section. Note that the mortar is lime rich. Width of sample = 60mm.

The mortar is well compacted and firm, and required light to moderate finger pressure to break, but once disrupted the sample could be powdered with relative ease. The mortar was found to be fully carbonated and there was no evidence in the sample examined of the presence of any fibrous materials or other organic additives.

The mortar was composed mainly of clay with a proportion of sand grains dominated by quartz, along with feldspar, limestone and shell fragments. The binder used is lime and there was no evidence to suggest the presence of any other form of binder or pozzolana in the mix. The limestone fragments are from a bioclastic limestone, as was some of the limestone that was burnt to produce the lime used as the binder, see following plates. The bulk of the mix is dominated by clay minerals, which are below the resolution of the stereo-microscope.



**Plate No. 3:** Image of thin section prepared from plaster sample. The blue colour is from the blue dye added to the impregnation resin in which the sample is embedded prior to cutting. The blue dye highlights the porosity in the mortar along with any voids, and cracks that may be present.



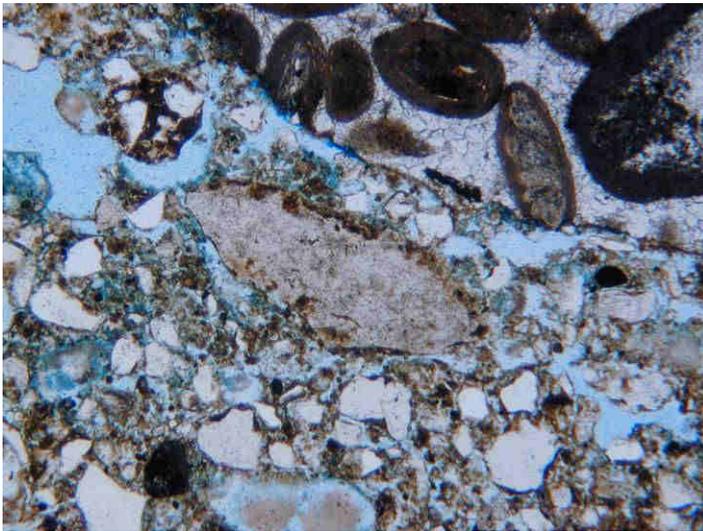
**Plate No. 4:**

Photomicrograph of the mortar fabric showing the general fabric.

The angular/sub-angular sand fragments are quartz grains. A partially hydrated lime inclusion can be seen on the right side of the plate, the inclusion is transacted by a crack.

The lower part of the plate is more porous and richer in lime than the upper part, which contains more clay and more dense.

Field of view 2.4mm in plane polarised light. Porosity is highlighted by blue dyed resin.

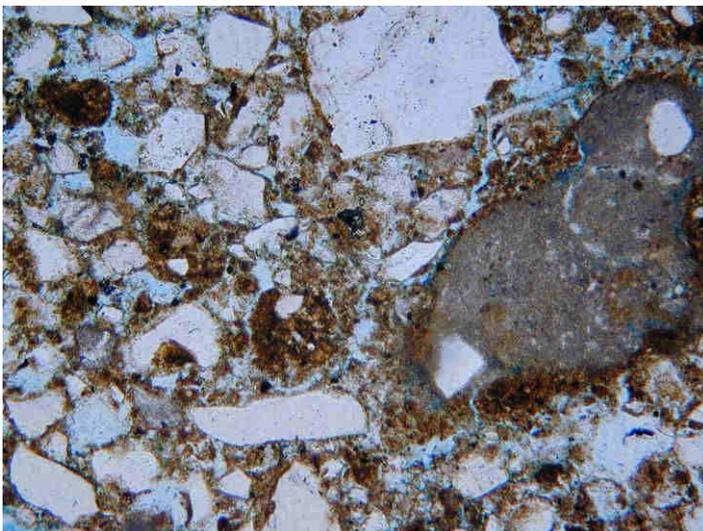


**Plate No. 5:**

In this view a bioclastic limestone fragment can be seen upper right, with a partially calcined limestone fragment in the centre and a fully hydrated lime putty inclusion at the lower edge, centre.

The mortar contains a large void in the upper left with an abundance of fine shrinkage cracks throughout the paste; this is contributing to the porosity in the mortar.

Field of view 2.4mm in plane polarised light. Porosity is highlighted by blue dyed resin.

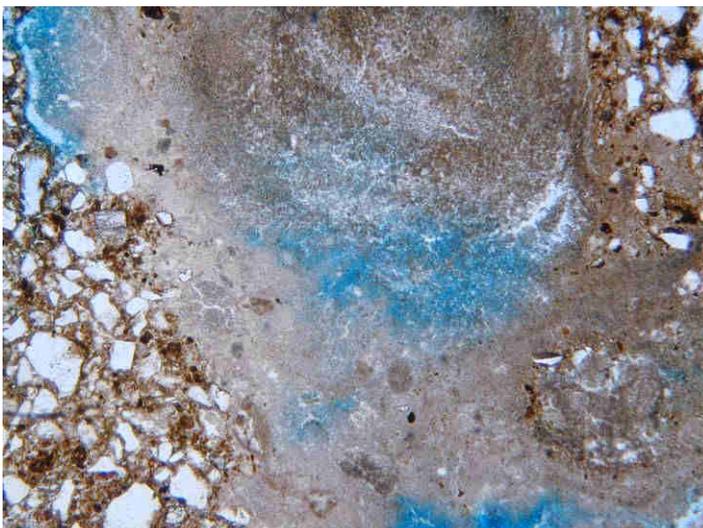


**Plate No. 6:**

The plate opposite shows another area within the mortar where a partially burnt limestone fragment can be seen in the right side of the plate, this retains the texture and structure of the original limestone.

Small clay nodules can be seen scattered throughout the mortar. Shrinkage cracks meandering through lime rich zones can also be seen, some have permitted fluid migration with leaching of binder, see centre of plate.

Field of view 1.6mm in plane polarised light. Porosity is highlighted by blue dyed resin.

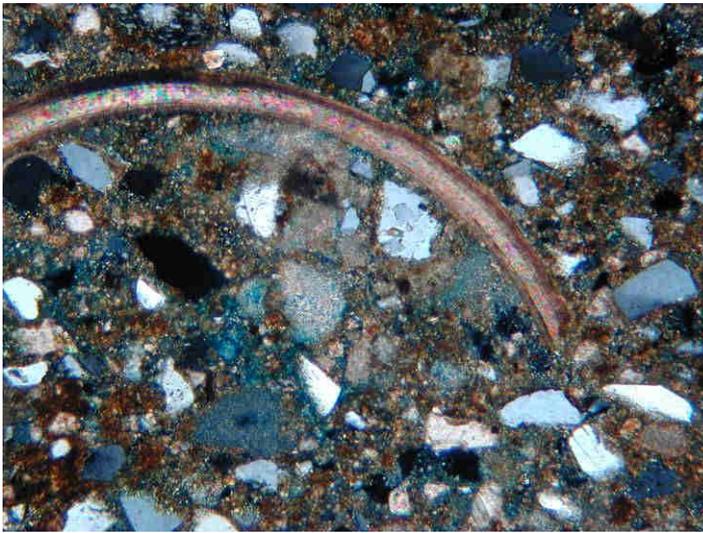


**Plate No. 7:**

This photomicrograph shows a large lime inclusion, where the lime has partially hydrated and is also diffusing into the body of the mortar, right side of plate.

The lower left is clay rich and contains an abundance of fine channel ways, some of which had transported lime rich fluids during mixing and placing.

Field of view 1.6mm in plane polarised light. Porosity is highlighted by blue dyed resin.

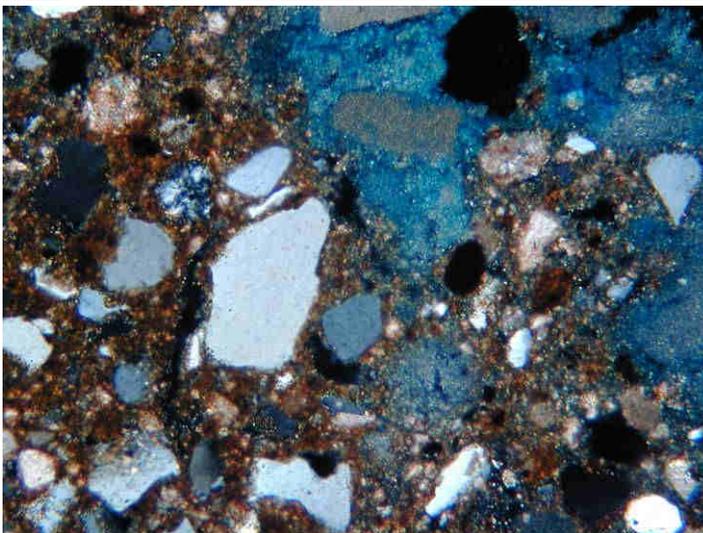


**Plate No. 8:**

This photomicrograph shows a shell fragment transecting the image, within a lime rich clay matrix, where the lime is finely diffused through the mortar.

The lime is fully carbonated and this is confirmed by the white speckling seen throughout the mortar in cross polarised light.

Field of view 2.4mm in cross polarised light.  
Porosity is highlighted by blue dyed resin.

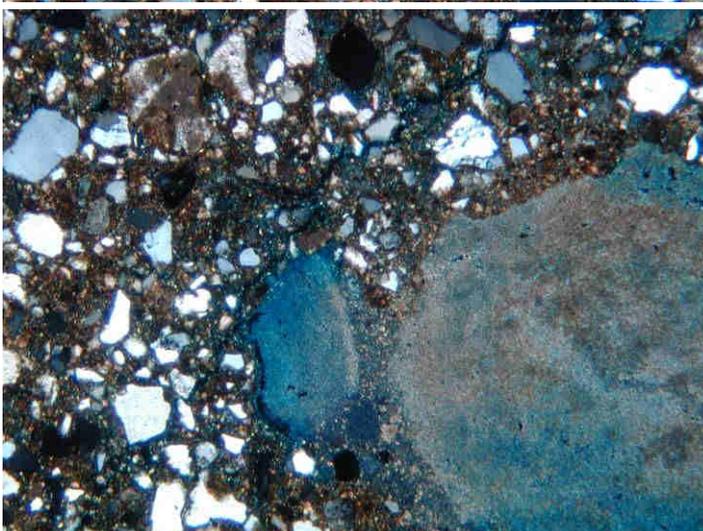


**Plate No. 9:**

A photomicrograph in cross polarised light of an area where a lime putty inclusion (right side of plate) is partially mixed into the adjacent clay rich fabric, left side of plate.

The majority of the sand grains in view (white and grey) are of quartz.

Field of view 1.6mm in cross polarised light.  
Porosity is highlighted by blue dyed resin.

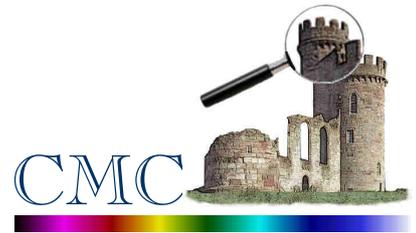


**Plate No. 10:**

A large intact lime inclusion can be seen in the lower right with a small separate inclusion in the lower centre.

The lime is carbonated and neither inclusion retains any rock fabric texture. The margins are distorted by the surrounding clay and sand grains, which in addition to their texture confirm that they were formed from putty rather than quicklime.

Field of view 2.4mm in cross polarised light.  
Porosity is highlighted by blue dyed resin.



## Results of Compositional Analysis

When the mix composition is determined simply from an analysis based on acid digestion alone, the mix proportion would be as follows:

1 part binder to 1.4 parts aggregate/clay by weight for a Putty lime mix,

or

1 part binder to 1.3 parts aggregate/clay by volume, for a Putty lime mix.

The following mix proportions would produce a similar mortar, if based on Quicklime mix:

1 part binder to 3.1 parts aggregate/clay by weight for a Quicklime mix,

or

1 part binder to 1.0 parts aggregate/clay by volume, for a Quicklime mix

However, as there is both limestone shell and lime inclusions to consider the mix composition was determined from a modal analysis on the basis of a 900 point count carried out on the prepared thin section. The mix corrected mix composition is therefore indicated to be as follows:

Proportion by mass, where the lime inclusions are included in the binder and the aggregate consists of the quartz, lithic fragments, limestone, shell and clay:

1 part binder to 3.5 parts aggregate/clay by weight for a Putty lime mix,

or

1 part binder to 3.3 parts aggregate/clay by volume, for a Putty lime mix.

The following mix proportions would produce a similar mortar, if based on Quicklime mix:

1 part binder to 7.9 parts aggregate/clay by weight for a Quicklime mix,

or

1 part binder to 2.6 parts aggregate/clay by volume, for a Quicklime mix

Proportion by mass, where the lime inclusions are included in the aggregate and the aggregate consists of the quartz, lithic fragments, limestone, shell and clay + lime inclusions, i.e. giving a measure of the effective lime content:

1 part binder to 8.0 parts aggregate/clay by weight for a Putty lime mix,

or

1 part binder to 7.5 parts aggregate/clay by volume, for a Putty lime mix.

The following mix proportions would produce a similar mortar, if based on Quicklime mix:

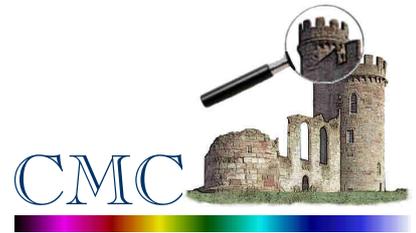
1 part binder to 17.9 parts aggregate/clay by weight for a Quicklime mix,

or

1 part binder to 5.8 parts aggregate/clay by volume, for a Quicklime mix

From a modal analysis it was determined that the sand to clay ratio was in the region of 1 part clay to 2.8 parts sand by volume.

The aggregates recovered from the acid digestion were graded through a nest of British Standard sieves with the following particle size distribution obtained; see the table below and the aggregate filled histogram presented in Figure No. 1.



Sample Reference	SR2211 – S1 Plaster sample	
	Percentage Retained	Percentage Passing
British Standard Sieve Size		
4.00mm	0	100
2.00mm	2.0	98.0
1.00mm	2.8	95.2
0.500mm	1.7	93.5
0.250mm	4.9	88.6
0.125mm	10.3	78.3
0.063mm	34.1	44.2
Passing	44.2	

### X-Ray Powder Diffraction

As it was necessary to determine the mineralogy of the binder to confirm if it displayed any hydraulicity a sample was prepared for analysis by XRD. This was achieved by picking the material from the core of a number of the soft lime inclusions, with the material ground in an agate mortar and pestle until it all passed a 63µm sieve. The prepared powder was dispersed onto a glass slide in the form of an acetone suspension which was evaporated to dryness in preparation for presentation in the diffractometer.

The sample was analysed in a Philips X-ray Diffractometer fitted with a single crystal monochromator, set to run over the range 3° to 60° 2θ in steps of 0.1° 2θ at a rate of 1° 2θ/minute using CuKα radiation. The digital output from the diffractometer was analysed by 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 of the analysis, by X-ray Diffraction, is presented in the following Figure in the form of a labelled X-ray Diffractogram:

**Figure No. 2** – Sample SR2211 – Plaster on the Hard from the Stair at York House.

The abbreviations used on the chart, to identify peak positions, are as follows:

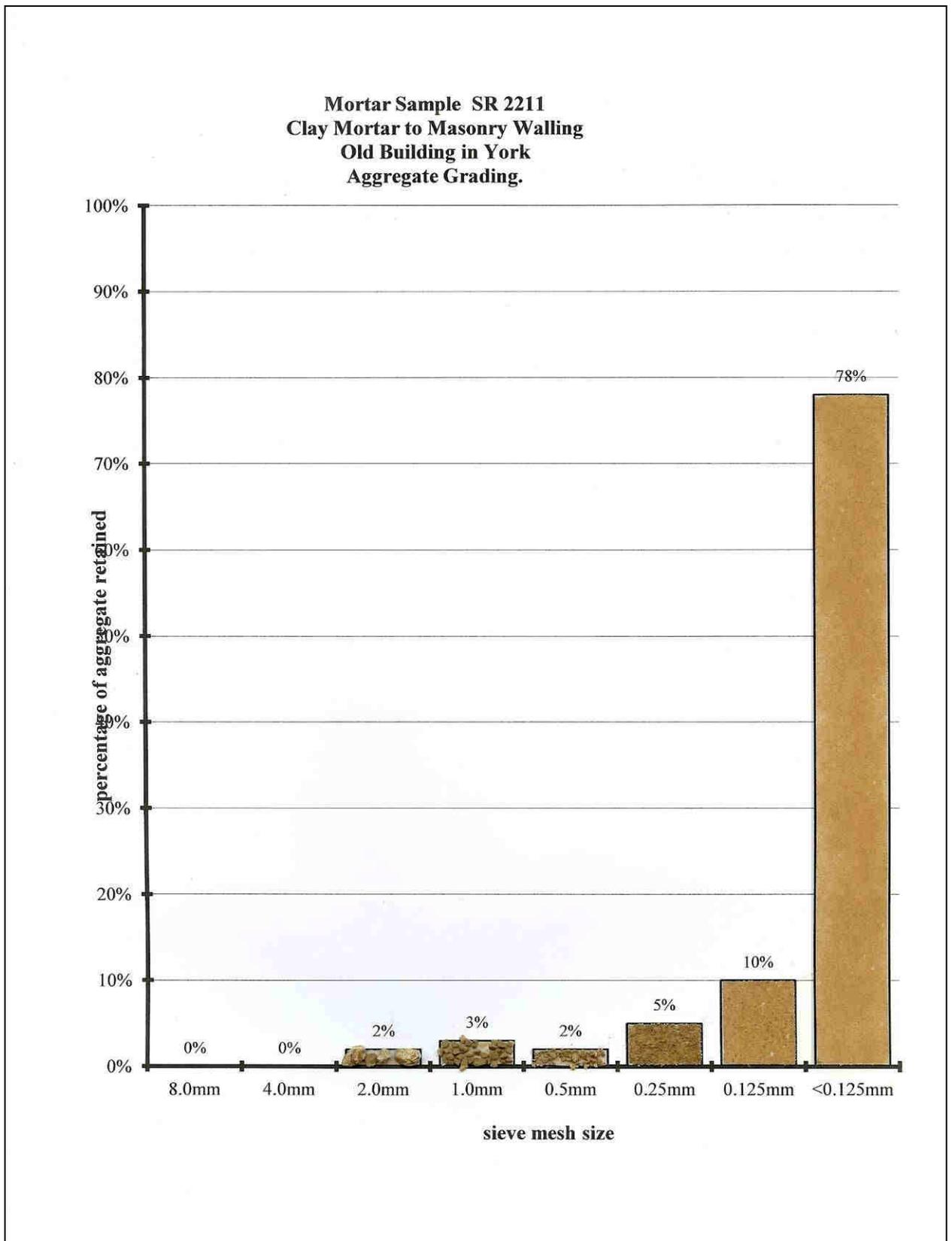
- cc** = Calcium carbonate (CaCO<sub>3</sub>), carbonated lime, major component of the lime binder,
- qz** = Quartz (SiO<sub>2</sub>), component of clay, possible contaminant from the mortar,
- fs** = Feldspar, various forms present but dominated by Albite and Anorthite, both of the Plagioclase group, natural mineral component commonly found in sands and clays.

On the basis of the XRD analysis it is indicated from the mineralogy of the lime, which is predominantly of calcium carbonate, that the lime was a non hydraulic high calcium lime at the point of use.

### Quality Statement

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

The results presented and comments offered relate only to the clay/lime plaster from the stair at York House received on the 17<sup>th</sup> December 2014 from Nigel Copsey.



**Figure No. 1:** Grading of Aggregate & Clay recovered from Plaster sample.

