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Our Ref: M/1921/18/C1
Your Re.: Clay Mortar

10th October 2018

CERTIFICATE OF ANALYSIS OF A MORTAR SAMPLE FOR DETERMINATION OF MIX COMPOSITION & GRADING ANALYSIS

Project Reference	:	Clay Mortar from Normandy
Sample Description	:	T/F Infill
Date Received	:	29th August 2018
CMC Sample Ref	:	SR 2621 – S1
Date Analysed	:	11th to 13th September and 9th & 10th October 2018
Method of Test	:	Mix composition with grading of recovered aggregate Binder type Identification by X-Ray Diffraction, with quantification analysis and thin section examination following standard petrographic procedures.

Sample

A sample of mortar identified as "Clay Mortar" was received in CMC's Stirling Laboratory on the 29th August 2018. The sample was delivered by Nigel Copsey of the Earth, Stone & Lime Company, with a request that the sample be submitted to a programme of examination and analysis to establish its composition, with particular emphasis on determining the presence of lime in the mortar.

To achieve this, the binder type was initially examined with the aid of a stereo-binocular microscope at magnifications up to x20. Following this a representative sub sample was prepared and analysed by X-ray Diffraction, which would provide further information on the type of binder employed in the production of the mortar, and would also help to identify if there were any deleterious reaction products present.

On receipt in the laboratory, the sample details were entered the sample register and the unique sample identification number SR2621-S1 allocated.

The sample details are presented below:

CMC Sample Ref.	Client Ref	Location Sampled
SR2621 – S1	T/F Infill, Normandy	Mortar from a clay mortared stone building in Normandy.

Method of Test

On receipt in the laboratory the sample was logged, with its mass and size recorded prior to being photographed, in the as-received condition. The sample was then submitted to an examination with the aid of a stereo-binocular microscope at a magnification up to x20 in preparation for analysis.

During the microscopic examination the sample was exposed to a series of *ad hoc* droplet tests employing a range of reagents and indicator solutions to aid the identification of the components present and to assess the condition of the mortars as received.

Following the initial examination, a representative sub-sample was obtained and this dried to a constant weight prior to being lightly ground and disaggregated in an agate mortar and pestle in preparation for determination of its mix composition.

In the event that it may have a proportion of lime present the mix composition of was initially determined by acid digestion following the procedures of the Scottish Lime Centre Trust (SLCT). On completion of the digestion, the aggregate in the sample was recovered by vacuum filtration, dried and graded through a nest of British Standard sieves.

Confirmation of the binder type used in the production of the mortar was confirmed by X-ray Diffraction (XRD) analysis. This was achieved by crushing and grinding a further representative sub-sample in an agate mortar and pestle, until it all passed a 63 micron sieve. With the material passing the 63micron sieve collected and back-packed into proprietary sample holders in preparation for presentation in the X-Ray Diffractometer.

A petrographic thin section was also prepared to permit the form in which the binder was used to be assessed, and if possible, determine other features of the mortar that may assist in its replication for use in conservation, restoration or repair works.

Observations from Macro/microscopic examination

The sample was logged on receipt with the following determined:

Sample Ref.	Client Ref.	Mass of Sample (gram)	Dimensions of Largest piece (mm)	Colour by the Munsell Soil Colour Charts
SR2621-S1	Clay mortar	156.6	79.5 x 74.9 x 39.2	10YR 7/6 “Yellow”

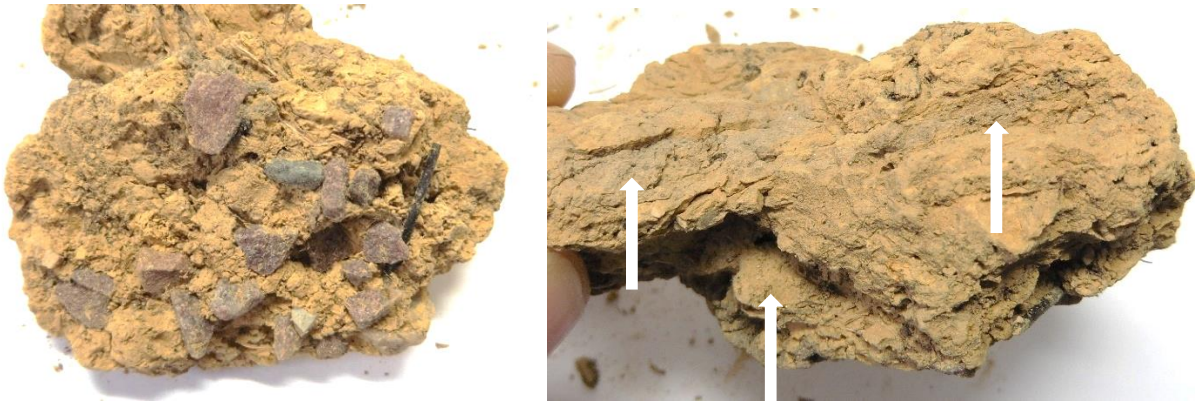
The sample received consisted of two large pieces of a well bound clay mortar and a small quantity of loose fines.

The mortar was well compacted but relatively soft and the intact pieces could be broken under light finger pressure and once disrupted the mortar was very easily powdered. The mortar in both pieces, although well compacted, was noted to contain an abundance of irregular shaped voids, which have the appearance of placing artefacts, due to air entrapped within the mortar as it was placed, and from subsequent drying.

The mortar is comprised of clay and a fine sand, which although generally well mixed, was observed to contain a number of thin lenses of unbound very fine sand grains. An abundance of chopped fibre was observed to be present, with this including straw and various grasses (stalks), with this locally appearing in clusters, albeit these are well distributed throughout the mix. The fibres range in width from 0.08 to 4.24mm and 4.16mm to 19.8mm in length, within the sub-sample examined.

One of the fragments retained small reddish aggregates embedded in one of its larger surfaces, possibly the outer surface. The chips were of a crushed pink granite and may be the residue from a surface coating (Dash aggregate, Harl or similar).

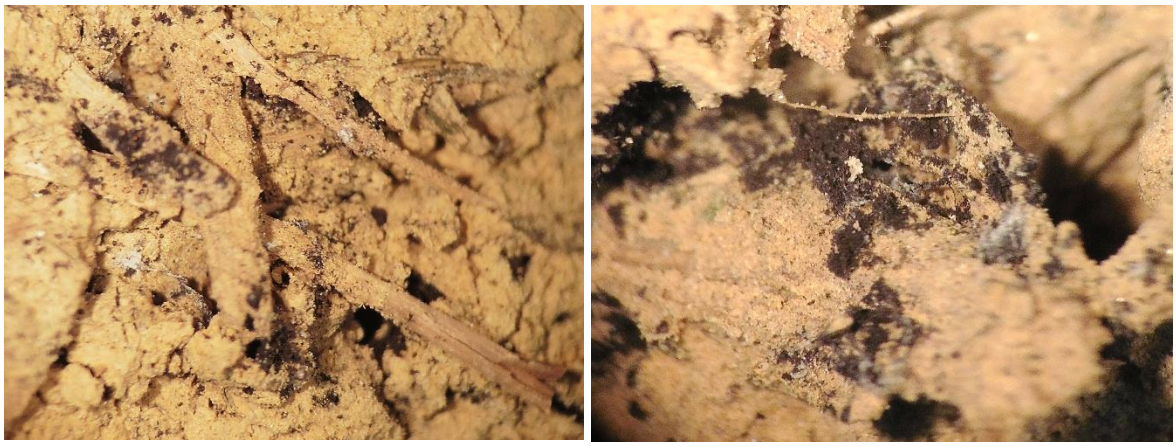
Plates No. 1 & 2: The above plates show the intact pieces of mortar as received, with the left plate showing one of the larger surfaces of the intact pieces, with the right plate showing the opposite face. Note the presence of a crushed aggregate chips in the surface of one fragment (right piece).



Plates No. 3 & 4: The left plate shows a close-up of the aggregate embedded in the surface of one of the pieces, with the aggregate appearing to be a crushed pink granite chip. The right plate shows a fractured edge where sandy lenses can be seen within the thickness of the mortar, these are more grey in colour, arrowed in the plate.

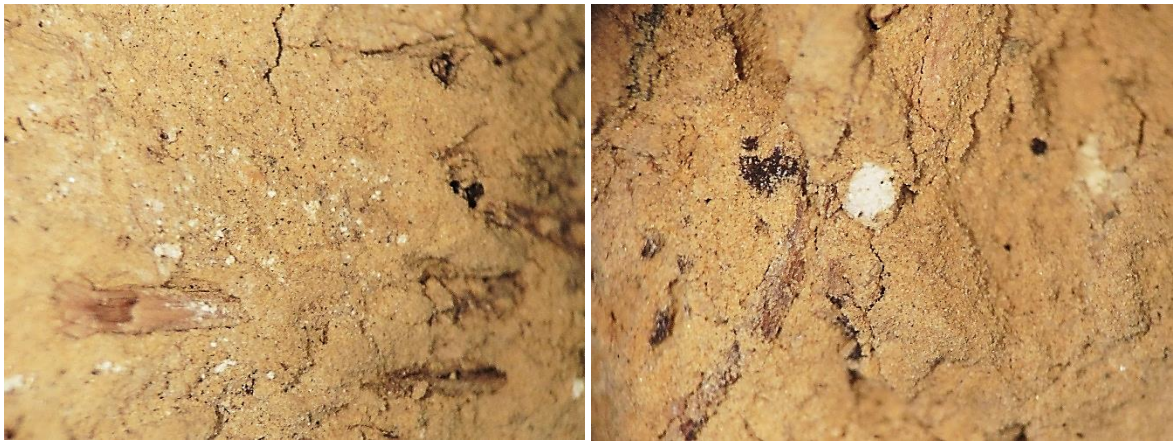
Plates No. 5 & 6: The above plates show fractured surfaces in which the abundance of fibre can be seen, with straw and grass stalks both apparent.

There was also small dark fragments and clusters randomly distributed throughout the mortar, which ranged in size from 0.06mm to 5.8mm. These had the appearance of globular masses, some of which contained clusters of very fine fibrous material, possibly indicative of insect activity, within the mortar. On examining these dark inclusions tended to disintegrate on probing and were very soft and appeared to possibly be organic in origin. Without further analysis it is not possible to conclusively identify this material, however, it is considered, given its occurrence in association with the straw and grasses, that it may be indicative of animal manure having been incorporated within the mortar.



Plates No. 7 & 8: The above plates show microscopic images. The left image shows of the fibre within the mortar, and the dark clusters commonly associated with these. The right plate is a magnified area adjacent to a fibre, where insect web material can be seen within the dark mass.

Lime inclusions were noted within the mortar, but these were sparse and very localised in occurrence, with that observed forming very small inclusions, typically less than 0.1mm in size but locally up to 0.7mm.



Plates No. 9 & 10: The above plates show examples of a rare location within the sample where lime was apparent, with the left plate showing fine inclusions of lime (white in image), with the right plate showing two of the larger inclusions, with the larger (right) inclusion measuring 0.7mm in diameter.

Results of XRD Analysis for Binder Type

A powdered sub-sample of the mortar was analysed in a Philips X-ray Diffractometer to aid in confirming the type of clay and any added binder used in the mortar preparation. The powdered sample was collected from material ground to pass a 63µm sieve, with this back-packed into a proprietary sample holder for presentation in the diffractometer.

The Diffractometer was 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 results of the analysis, by X-ray Diffraction, are presented in the following attached Figure, in the form of a labelled X-ray Diffractogram:

Figure No. 2: Sample SR2621-S1, Powdered sample of clay mortar ex Normandy.

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

cc = Calcite ($CaCO_3$) calcium carbonate, carbonated lime from lime binder, or limestone,
qz = Quartz (SiO_2) dominant component of the sand aggregate in the mortar,
fs = Feldspar, common rock forming minerals, with mostly Albite and Microcline identified,
pe = Penninite, layer-lattice clay mineral of the chlorite group of minerals,
mi = Muscovite mica, common rock forming mineral, commonly found in clays.

The data from the XRD Analysis was processed further by Rietveld refinement to enable quantification of the crystalline components present, with the following results:

Component	**Proportion (% by Mass)**
Sample:	**Mortar sample**
Calcite	2.5
Quartz	75.9
Feldspar (Albite)	10.8
Feldspar (Microcline)	3.1
Penninite	4.1
Muscovite	3.6
Total	100.0

Based on the XRD analysis, it is indicated that the mortar is essentially a clay sand mix, with a trace proportion of lime, with the lime being a high calcium lime. This analysis procedure does not determine the presence or proportion of organic components present in the sample analysed.

No reaction products or potentially deleterious components were detected in the sample analysed.

Mix Composition

The result of the composition analysis, determined by acid digestion, are presented below:

Sample Ref. No.	**SR 2621 – S1**	
Lime type (from XRD)	Non Hydraulic Lime	
Binder form:	Quicklime	Putty Lime
Lime Binder/Aggregate Ratio	1.0 : 39.3	
Weight proportions calculated mix ratio by dry mass.		
Lime	1.0	1.0
Aggregate	70.0	31.4

Approximate volume Proportions calculated on the basis of the standard data for a Non-Hydraulic lime.

	Quicklime	Putty Lime
Lime	1.0	1.0
Aggregate	22.2	25.3

The aggregates from the acid digestion was recovered and the particle size distribution determined, with the results presented in the table below and in the aggregate filled histogram in figure No. 1.

Sample Reference	**SR2621 – S1**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100
4.00mm	0	100
2.00mm	0	100
1.00mm	0.4	99.6
0.500mm	0.6	99.0
0.250mm	1.1	97.9
0.125mm	1.1	96.8
0.063mm	2.3	94.5
Passing	94.5	

Table No. 1: Results of the grading of recovered aggregate.

The aggregate in the mortar is dominated by silt and fine grained sand along with minor coarser quartz grains (<2.0mm) with feldspar (<0.5mm).

In an attempt to determine the quantity of fibre present a piece of the mortar was gently disaggregated and the straw and other fibrous material picked from the mortar and weighed, with the following result:

Proportion of fibrous material 2.8% by dry mass[1]

[1] Note the fibre is desiccated and very friable, therefore, this proportion may bear little relevance to the proportion of straw actually added at the time of mixing.

Microscopic Examination

To further clarify the form in which the binder was used a petrographic thin section was prepared from an intact piece of the mortar. This was impregnated with a blue dyed resin, one side of which was cut and polished prior to being mounted on a glass slide (50mm x 75mm), with the sample aligned to give the maximum area on the slide. The sample was then cut and polished to give a thickness in the region of 30µm in preparation for examination in the polarised light microscope.

Observations from the examination are presented below:

Aggregate

The aggregates in the mortar sample are dominated by quartz grains with chert and other lithic fragments, mostly of altered granitic rock types, also present.

The aggregate grains are predominantly sub-angular, with a majority of the grains displaying sharp margins, infering that the sand was not water transported. The aggregate grains range in size from 0.01mm to 0.72mm (very fine silt to medium sand) with rare coarse grains, up to 3.3mm in size, with these mostly of quartz and chert. There is a significant proportion of clay in the mortar, and this is counted as the binder.

A proportion of straw and other vegetative matter was included in the mortar, along with an amorphous material, which although not always observed in combination with the fibre, there is a high incidence of them both being observed together. It would be necessary to use other analytical techniques if it were necessary to clarify the form and source of the opaque materials present.

Binder

The binder is clay with a trace proportion of lime, the latter is very low, and it may be that it is included in the mortar as a contaminant, i.e. from a surface coating, rather than having been added to the mortar. There is, however, given its low proportion and its distribution, in one piece of the mortar to infer that it may have been added, to some of the clay, in the form of a quicklime as a dryer.

Voids and microcracks

Voids and cracks are present and appear to have formed in response to air entrapped during mixing and placing, drying shrinkage and from the decay of organic components. Voids range from 0.2mm to 1.3mm in width and up to 4.4mm in length. There is good connectivity between the voids. All voids are free of linings or secondary minerals.

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

Sample Ref:	**SR2621-S1**
Constituents	**%**
Quartz	52.2
Lithic fragments	7.1
Opaque	8.7
Fibre	13.6
Total Aggregate	**81.6**
Binder (Clay)	17.5
Lime	0.9
Secondary Minerals	0
Total Binder	**18.4**
Total Constituents	**100.0**
Cracks/Voids	**11.6**
Binder: Aggregate Ratio	**1.0:4.46**
Clay: Sand :Straw % by Vol	**18 : 68 : 14**

Table No. 2: Result of modal analysis (900-point count) on thin section

Photomicrographs:

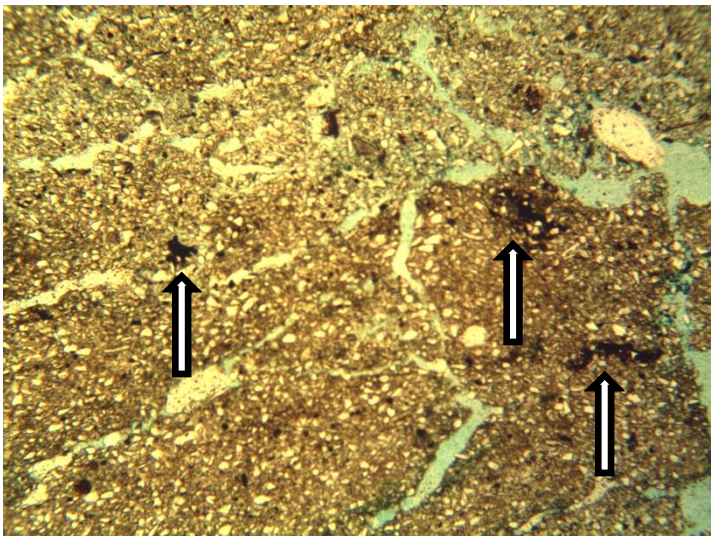


Plate No. 11:

A view in plane polarised light (ppl) showing a typical area of the mortar, with the area in view being dominated by fine sand bound in a clay matrix. The area in view is transected by an abundance of elongated voids and shrinkage cracks, not uncommon in clay mortars.

The dark areas, arrowed, appear to be concentrations of an amorphous material, within which some sand grains can also be seen, these are perhaps organic in form.

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

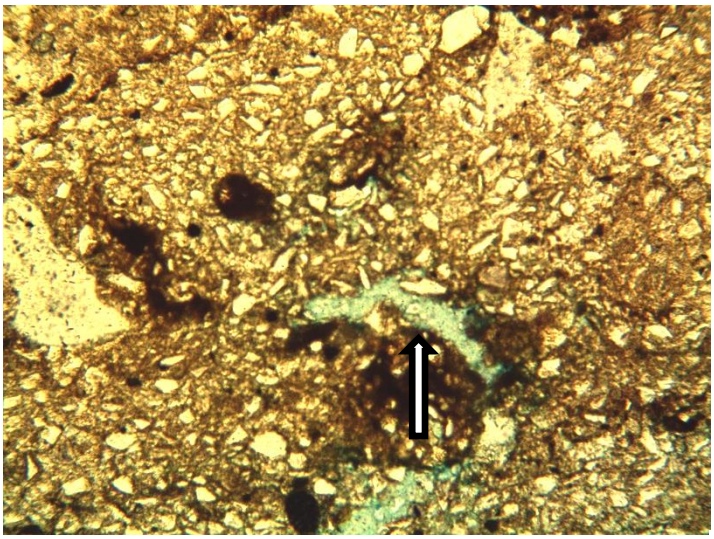


Plate No. 12:

A magnified view of another area of the mortar, again in ppl.

In this view there are some larger lithic fragments, centre left and upper right (chert). There is also a void formed from the decay of a fibrous material, arrowed in plate. Concentration of opaque material is also present in this view, and again these contain sand grains, inferring that it was of a plastic consistency when incorporated within the mortar.

Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

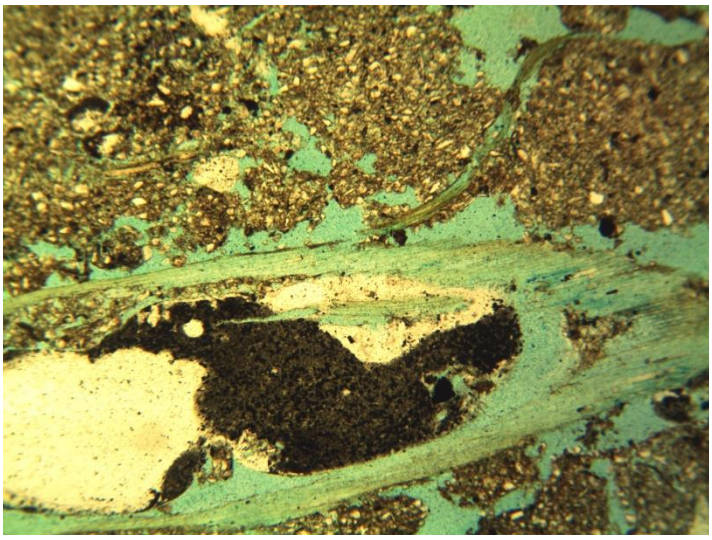


Plate No. 13:

Another view in ppl, of an area incorporating a disrupted wide straw fibre, which is fully encapsulated within the mortar. Note the concentration of opaque material in close proximity, and below the fibre. With it exposed where the fibre was cut during section preparation. The fibre had also started to separate during mixing, see upper edge.

Note the abundance of voids in the upper part of the plate

Porosity and voids are highlighted by the blue dyed resin. Field of view 2.4mm.

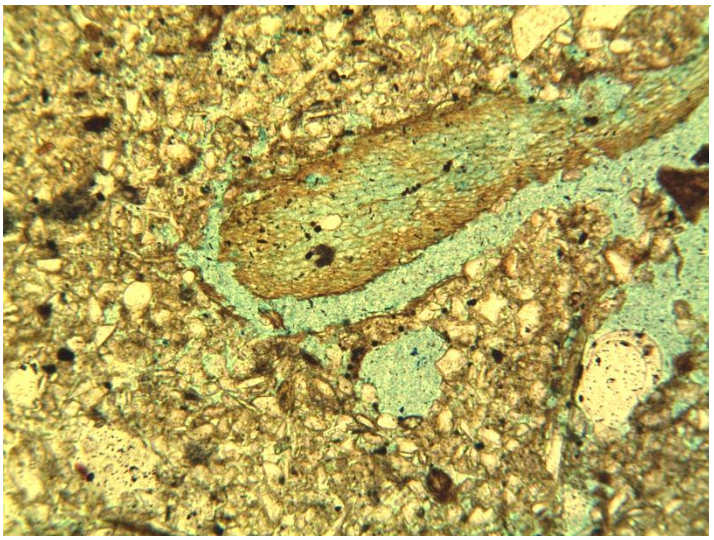


Plate No. 14:

Another view in Plane Polarised Light (ppl), showing a fibre cut at an angle to the plane of the slide.

There is a longitudinal void in the paste adjacent to the fibre indicating that some shrinkage, most likely in the fibre, had occurred after placing. The remainder of the mortar is relatively dense and typical of the mortar in the sample examined.

Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

Summary

From the examination and analysis of the mortar sample from the clay mortar T/F Infill sampled in Normandy, it is indicated that the mortar was made from clay sand mortar to which a straw fibre had been added. Albeit that the presence of an organic component was also noted to be present and it may be that a proportion of the fibre was incorporated along with animal manure as a binder/plasticiser in the mortar.

Details of the mix used is summarized below:

Sample Ref.	**SR2621-S1**
Volume Proportion by	***Modal Analysis***
Binder content (Clay)	1.0
Fine Aggregate (Sand)	3.7
Fibre	0.7

The aggregates in the masonry mortar is a fine to very fine quartz rich sand, with trace lithic grains.

The fibre is mostly of straw with a proportion of other grasses (stalks) also present.

Sample Reference	**SR2621 – S1**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
2.00mm	0	100
1.00mm	0.4	99.6
0.500mm	0.6	99.0
0.250mm	1.1	97.9
0.125mm	1.1	96.8
0.063mm	2.3	94.5
Passing	94.5	

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 sample of Clay mortar delivered to CMC's laboratory on the 29th August 2018 by Nigel Copsey of the Earth, Stone & lime Company, which was identified as Clay mortar from Normandy.

W A Revie
For CMC Ltd.

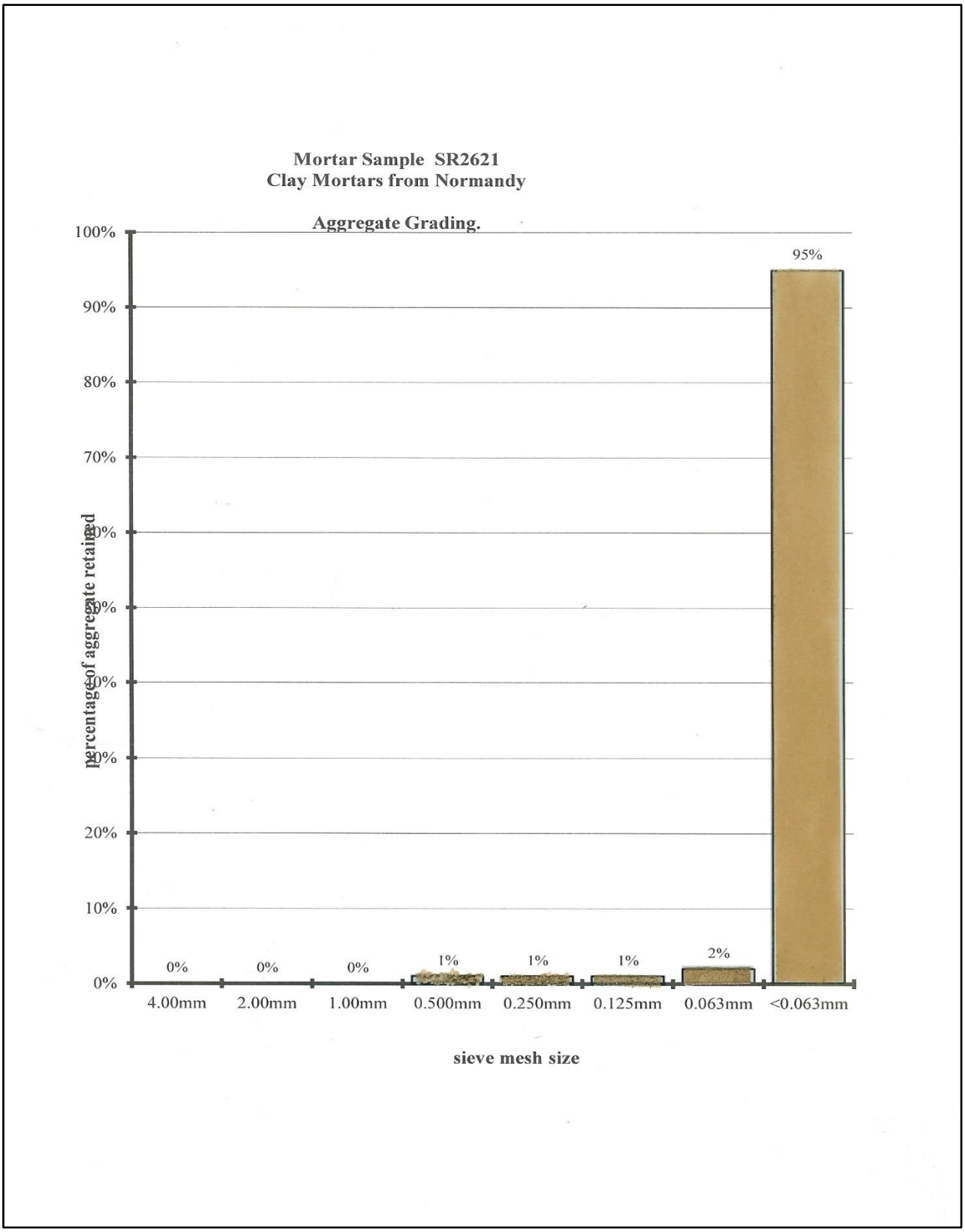


Figure No. 1 – Grading of recovered aggregate from Sample SR2621-S1

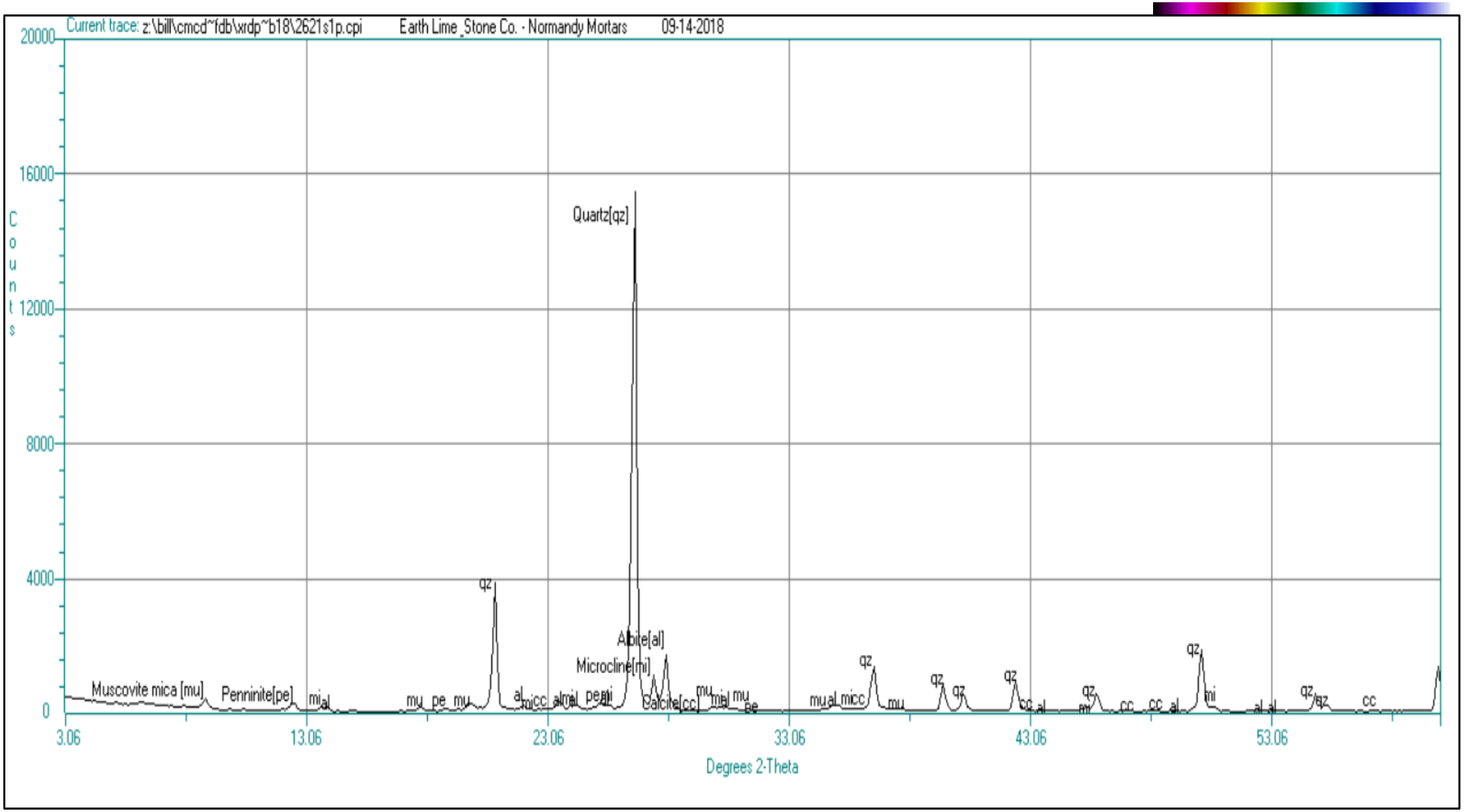


Figure No. 2: Sample SR2621-S1, Powdered sample of clay mortar ex Normandy.