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Our Ref: M/2098/21/C1
Your Ref.: 60 Goodramgate

4th February 2021

CERTIFICATE OF ANALYSIS ON A MORTAR SAMPLE FOR BINDER TYPE DETERMINATION & MIX COMPOSITION

Project Reference	:	60 Goodramgate, York
Sample Source	:	Brickwork Masonry from Eaves Level
Sample Description	:	Bedding mortar
Date Received	:	21st January 2021
CMC Sample Ref	:	SR 2824 - S1
Method of Test	:	Mix composition by acid digestion, and binder type by XRD analysis by in-house procedures, with fabric condition from a thin section examination.

Samples

A sample of brickwork masonry, identified as having been sampled from eaves level on the 19th century brickwork building at 60 Goodramgate, York, was received in CMC's Stirling laboratory on the 21st January 2021. The sample was supplied by Nigel Copsey of the Earth, Stone & Lime Company.

CMC were advised, by email, on the 20th January 2021 that the sample was being despatched and requesting that it be submitted to examination and analysis to establish the composition of the mortar, identify the binder type used and assess its condition, and provide any other information that may be gleaned from the laboratory programme.

On receipt in the laboratory, the sample details were entered into the sample register and the unique sample identification number SR2824 allocated. The details of the sample received is reproduced below:

CMC Ref.	Client Ref	Location Sampled
SR2824 - S1	Masonry Mortar	Brickwork masonry at eaves level on 60 Goodramgate, York.

Method of Test

The sample was photographed on receipt, with its mass and size recorded prior to it being submitted to an examination with the aid of a stereo-binocular microscope at a magnification up to x10. During the 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 mortar sample as received.

To permit confirmation of the type of binder used in the production of the mortar a representative binder rich, sub-sample was prepared for analysis by X-ray Diffraction (XRD). The sub-sample was initially prepared by disrupting the sample in an impact mortar, then disaggregated the material further by gently grinding it in an agate mortar and pestle. This was carried to separate the binder from as much of the aggregates as possible, with a binder rich sub-sample recovered by sieving the disaggregated material over a 63µm sieve.

The sample was prepared for analysis by backpacking the powder into a proprietary sample holder in preparation for presentation in the diffractometer. With the specimen 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°.

A further sub-sample was dried to a constant weight, and the as-received moisture content determined. With the dried material then disrupted and lightly ground in a mortar and pestle in preparation for digestion of the binder in dilute hydrochloric acid. On completion of the acid digestion the residue was recovered by vacuum filtration, dried and aggregate graded through a nest of British Standard sieves.

To permit further information to gleaned from the sample, an intact piece of mortar was selected and used in the production of a petrographic thin section. This to assist in establishing the form in which the binder was used in the production of the mortar.

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)	No. of Pieces	Colour by the Munsell Colour Charts
SR2824-S1	Mortar	103.0	40.6 x 37.5 x 23.3	3 No	2.5Y 8/1 “White”

The sample consisted of three intact pieces of mortar all of which contained lime inclusions, which were noted to measure up to 7.3mm in size. The inclusions were mostly irregular though some were also noted to be sub-round to round in shape.

The mortar was moderately hard and well compacted with a low entrapped void content indicated. The voids appeared to be free of any redeposited material, or soiling, suggesting that water percolation through the mortar had not occurred, or was minimal.

The as-received moisture content was determined and found to be 1.9%, by dry mass.

The mortar is moderately hard, but could be broken under moderate finger pressure, and once disrupted it could be powdered further under light to moderate finger pressure. Aggregates could be picked from the fractured surfaces under light finger nail pressure.

On testing a freshly fractured and sawn surface with a phenolphthalein indicator solution, the mortar was found to be fully carbonated, with no colour change in the indicator solution observed.

From a water droplet test on freshly fractured and bed surfaces, it was confirmed that the mortar pieces all contained a well-connected pore structure, with the droplets, placed rapidly absorbed and quickly diffused through the mortar to depth.

Plate No. 1: The above plate shows the three pieces of mortar in the sample, as received. Note the presence of lime inclusion, irregularly distributed throughout the fabric. Soiling was evident, on the edge of one fragment, which was considered to be an outer joint face, see right particle in the plate.

Plates No. 2 & 3: The above plates show freshly fractured surfaces, from two of the fragments. Lime inclusions can be seen in both pieces, with the inclusions having the appearance of having been formed from either a quicklime, putty lime, or both.

The aggregates within the body of the mortar had the appearance of natural fine grained sand, dominated by quartz grains along with minor lithic fragments, with a maximum particle size of 3.4mm in the sample examined. The aggregates were well distributed, but grain to gran contacts were noticeably low, and the mortar appeared to be binder rich.

Results of XRD Analysis

To confirm if the binder was a lime, and if so, if there were any hydraulic components present, a binder rich sub-sample was prepared and submitted to analysis by X-ray Diffraction (XRD).

The results of the analysis are presented in the following figure, in the form of a labelled X-ray Diffractogram:

Figure No. 2: Sample SR2824-S1 – Binder rich fines from the bedding mortar sample.

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

cc = Calcite ($CaCO_3$) calcium carbonate, carbonated lime from binder, and this can also be present in any limestone the aggregate,

va = Vaterite ($CaCO_3$) another form of calcium carbonate, carbonated binder from lime binder, or in limestone aggregate, and also found in redeposited leached lime,

be = Belite (Ca_2SiO_4) *di*-Calcium Silicate, a hydraulic clinker component, common in Portland type cements and many hydraulic limes,

al = Alite (Ca_3SiO_5) *tri*-Calcium Silicate, another hydraulic clinker dominant in Portland cements and common in eminently hydraulic limes,

qz = Quartz (SiO_2) dominant component of the aggregate minerals in the sand,

fs = Feldspar, mostly of Albite of the Plagioclase group, with Sanidine, an alkaline feldspar, present as aggregate components,

do = Dolomite, ($CaMgCO_3$) present as a component of dolomitic lime or in the aggregate within any dolomitic limestone present,

ka = Kaolinite, clay mineral from the weathering/destruction of feldspar, present in the aggregate.

On the basis of the XRD analysis, it is indicated that the mortar sample was made from either a hydraulic lime or a High Calcium Lime/Hydraulic Lime blend, or a lime/cement binder. The mortar is fully carbonated, with no unhydrated lime present, and there was no evidence of the mortar having been affected by a reaction with any form of environmental sulphate, or other deleterious contaminant.

The data from the XRD analysis was processed further by Rietveld Refinement, in the Maud computer program. This was to permit quantification of the minerals and crystalline material present. See the results below:

Sample Ref.	**SR2824-S1**
Material	**Concentrated Matrix Fines**
Component	**Proportion (% by Mass)**
Calcite	59.5
Vaterite	0.4
Belite	0.6
Alite	4.1
Quartz	32.6
Feldspar (Albite)	0.1
Feldspar (Sanidine)	0.1
Dolomite	1.1
Kaolinite (Clay)	1.5
Total	**100.0**

From the above it is confirmed that the sample contains hydraulic components, due to the binder containing both Belite and Alite. However, the ratio of the hydraulic components detected are such as to suggest that the binder was most likely a Hydraulic Lime/High Calcium Lime blend, rather than a Portland type cement and lime binder.

Mix Composition

To determine the mortar mix composition and permit the recovery of the aggregate, for grading analysis, a further representative sample was prepared and submitted to determination of mix composition by acid digestion.

The result of the composition analysis carried out is presented below:

Sample Ref. No.	**SR 2824-S1**	
Binder type (from XRD)	Non-Hydraulic Lime	
	Quicklime	Lime Hydrate
Lime: Aggregate Ratio	1.0 : 2.3	
Weight proportions calculated mix ratio by dry mass.		
Lime	1.0	1.0
Aggregate	2.8	2.6
Approximate volume Proportions, calculated on the basis of the standard data for a non-Hydraulic Quicklime and a lime Putty		
Lime	1.0	1.0
Aggregate	1.5	1.8

The aggregate from the acid digestion was recovered, washed, dried and sieved through a nest of standard sieves. The particle size distribution was determined with the results of the aggregate grading presented both in the form of an aggregate filled histogram, in the appended figure No. 1 and in tabular form below:

Sample Reference	**SR2824 – S1 Brickwork Mortar**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100
4.00mm	0	100
2.00mm	0.9	99.1
1.00mm	9.4	89.7
0.500mm	52.5	37.2
0.250mm	28.6	8.6
0.125mm	4.4	4.2
0.063mm	3.3	0.9
Passing	0.9	

Table No. 1: Grading analysis of recovered aggregate, following acid digestion

The aggregate particles are dominated by quartz grains, with minor sandstone, limestone, quartzite, and altered igneous rock fragments, along with trace proportions of feldspar and opaque minerals. The particles are sub-angular to sub-round in shape.

The low proportion passing the 63-micron sieve, and the single size grading, may infer that the sands were obtained from a river, river bank or river terrace source.

Microscopic Examination

To clarify the condition of the fabric of the mortar and offer comment on the form in which the binder was used, a petrographic thin section was prepared from an intact piece of the mortar. With this subjected to examination in the polarised light microscope.

Aggregate

The aggregates are dominated by quartz grains along with a proportion of lithic fragments present. The latter are composed of quartzite, chert, sandstone, and limestone, along with trace proportions of opaque minerals. Although it is not possible to clearly identify all of the opaque minerals by optical microscopy alone, ironstone and coal are both considered to be present.

The aggregate grains, are typically sub-angular to sub-round in shape, and locally elongated, with many displaying water worn margins. The shape, apparent single size, and the texture of the aggregates suggesting a fluvial source for the sand, which may be from a local water course.

The sand grains range in size from 0.07mm to 2.85mm and are dominated by quartz grains, with minor quartzite, and chert, altered igneous rock types and weathered sandstone fragments present along with rare coal and ironstone particles. The aggregate would be classed as a medium grained sand, with very low fines content, i.e., <0.2mm> This would have resulted in a harsh mortar requiring a high binder content to be workable. This is confirmed in the thin section with the mortar appearing to be binder rich with all aggregate grains fully surrounded, and separated, by paste with grain-to-grain contacts being rare.

Binder

The binder is dense and contains lime inclusions, which are variable in appearance, with the inclusions showing features consistent with both quicklime and putty, along with balled hydrate. It is possible that the mix was batched from a quicklime that was slaked to a hydrate whilst mixed with the sand. With the mortar later remixed cold to achieve workability, during which a proportion of the binder took-up sufficient free water to form the putty globules observed.

Although a proportion of the inclusions display sharp outer margins, all were observed to be completely slaked, with a proportion of these also noted to display dolomitic features, and it is possible that the limestone used in the lime production contained a proportion of dolomitic, or magnesian limestone. The quicklime including the dolomite/magnesian lime would not slake as readily as that from the calcium limestone and this, it is considered, to a degree, is the reason for the variable condition of the inclusions. However, all of the inclusions are fully carbonated.

The paste is fully carbonated throughout the sample and there is no evidence of leaching, with loss of lime and the paste looks well compacted and in a sound condition.

The lime inclusions observed ranged in size from 0.2mm to 4.8mm in the section examined, but mostly these are <3.0mm. There is no residual rock fabric apparent in any of the inclusions and the lime appears to have been well calcined, though the hydration ranges from complete to patchy, the latter observed in the dolomitic/magnesian inclusions.

Hydraulic clinker is apparent, but very patchy in occurrence, with no relic grains observed within any of the lime inclusions, with the clinker only apparent as discrete grains, locally in clusters, distributed within the paste. It is, therefore, considered that a proportion of hydraulic quicklime was added to the mix, in the form of a hydrate, rather than infer that a proportion of the quicklime had been hydraulic. However, this is only an assumption, based on the observations from the condition of the slide examined.

There is, however, the possibility that there may have been some hydraulic quicklime in the mix, from burning limestone containing clay/silt laminations, but this would require additional samples and thin section examination to clarify.

Voids and microcracks

The voids observed in the mortar are mostly rounded to spherical in shape, and are predominantly entrained air voids. The voids range in size from 0.05mm to 0.2mm in size and occur in both in clusters and as discrete features, a common feature of overmixed wet mixes, or one to which an entraining agent had been added. All voids are free of linings or infillings with secondary minerals.

Cracks are minor, with those present having the appearance of early drying shrinkage cracks, and these are mostly short discreet cracks, up to 0.7m in length. These appear mostly to connect aggregates to voids, and voids to voids, providing a very fine connected pore structure throughout the paste. The crack margins are generally dense with the cracks have not acted as fluid migration channel ways. Cracks range in width from <0.02mm to 0.08mm.

Modal Analysis

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

Sample Ref:	**SR2794-S1**	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	31.6	31.6
Lithic Fragments	3.6	3.6
Sandstone/siltstone	6.4	6.4
Opaque Coal + Ironstone	0.7	0.7
Quartzite	4.4	4.4
Limestone	3.2	3.2
Lime inclusions	-	5.2
Total Aggregate	**49.9**	**65.1**
Binder (Lime)	37.6	37.6
Lime inclusions	5.2	-
Clinker	7.3	7.3
Secondary products/Calcite	0	0
Total Binder	**50.1**	**44.9**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**16.3**	**16.3**
Binder: Aggregate Ratio	**Total**	**Effective**
	1.0 : 1.0	**1.0 : 1.45**

Table No. 2: Result of modal analysis (600-point count) on the thin section from SR2724-S1.

The mix proportions determined from the modal analysis are reported as both Effective and Total binder content to aggregate ratio, by volume.

The effective binder content determined from the modal analysis is calculated on the basis that all of the lime inclusions, that have not diffused into the paste, are acting as aggregate rather than 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 composition at the time the mortar was made and placed, including all of the inclusions as part of the added lime binder, and this reflects the mix proportioning at the time of mortar was prepared.

Photomicrographs:

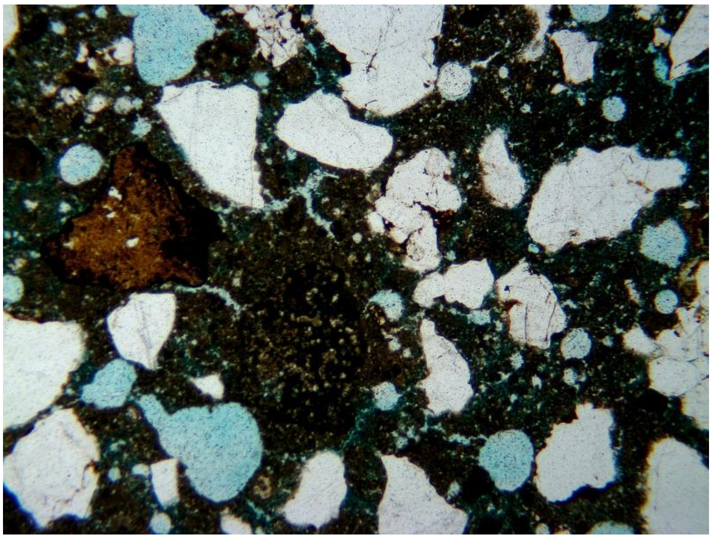

Plate No. 4:

A view in plane polarised light (ppl) of a typical area of the mortar, showing a very dense fabric contains few fine cracks. The aggregates are fully encapsulated in the fully carbonated lime paste, and are dominated by quartz grains (white in the plate). There are several small lime inclusions apparent in this view, lower centre and centre left. The inclusion although completely calcined and fully slaked do not diffuse into the surrounding paste, and act as aggregates. Note abundance of small rounded air voids

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

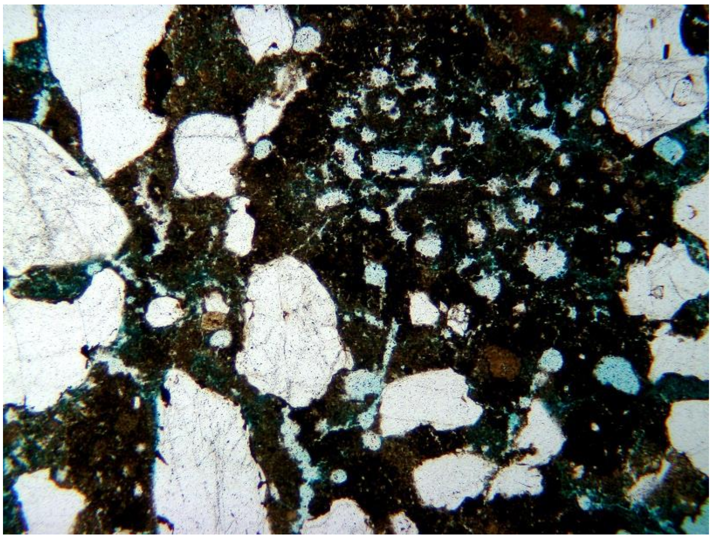

Plate No. 5:

Another view in plane polarised light, ppl, where a large lime inclusion can be seen filling the upper right quarter of the plate, the inclusion has been partially slaked and displays partially dolomitic features. This contains an abundance of open pores and displays a compact rim around its margin, indicating late slaking. Irregular clinker grains can be seen distributed around the surrounding paste. The aggregates in view are dominated by quartz grains, with minor opaque minerals and lithic fragments.

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

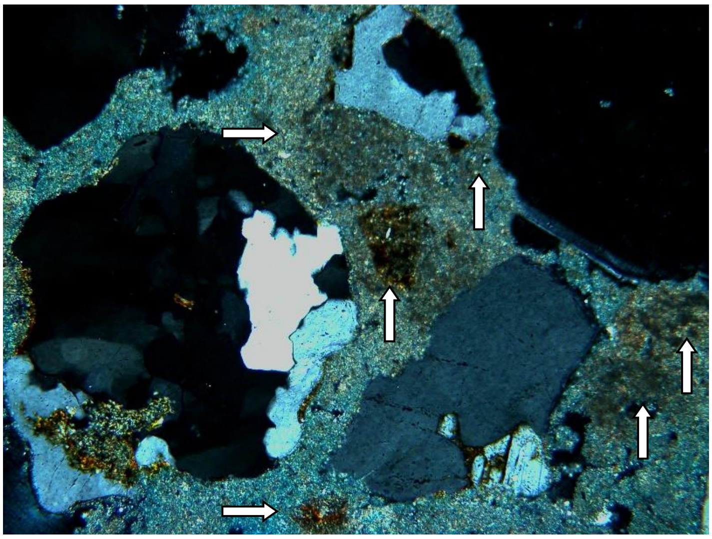

Plate No. 6:

A view in cross polarised light (xpl), of a dense area of paste in which an abundance of partially hydrated clinker grains can be seen, arrowed in plate. The grains are noticeably coarse. The paste in the mortar is carbonated, with that at the margin in the plate displays patchy microporosity. Aggregates are again dominated by quartz with minor opaque minerals. Voids are present in the upper left and right.

Porosity, voids, impregnating resin and opaque minerals all show dark in xpl. Field of view 1.2mm.

Summary

From the examination and analysis of the mortar sample from the Brickwork at 60 Goodramgate, York, it is indicated that the mortar was made from a natural single sized river sand and a quicklime binder containing both high Calcium and Dolomitic lime. There is a degree of hydraulicity in the mortar, though from this examination this is not considered to be from the quicklime, with it inferred that a proportion of a hydraulic lime hydrate had been added to the mix. The mortar shows all the features of having been mixed as a Hot Mixed Mortar, with this slaked to a hydrate prior to being remixed and used in the form of a cold mortar.

The addition of a small proportion of hydraulic lime hydrate during the second mixing is likely, with the proportion of hydrate added being at a dosage in the region of 1 part quicklime to ¼ part hydraulic hydrate (moderately hydraulic lime). Details of the mix composition is summarised below:

Sample Ref.	**SR2824- S1**	
Volume Proportion by acid digestion	***Quicklime***	***Hydrate***
Binder content (Lime)	1.0	1.0
Fine Aggregate (Sand)	1.5	1.8
By modal analysis		
Binder: Agg. by vol. (Total)	1.0 : 1.0	
(Effective)	1.0 : 1.5	
Quicklime : Hydraulic Hydrate: Sand	1.0 : 0.25 : 2.0	

The aggregates in the mortar are a natural quartz rich sand, with minor lithic fragments.

Sample Reference	**SR2735 – S1 Wall Core Mortar**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
4.00mm	0	100
2.00mm	0.9	99.1
1.00mm	9.4	89.7
0.500mm	52.5	37.2
0.250mm	28.6	8.6
0.125mm	4.4	4.2
0.063mm	3.3	0.9
Passing	0.9	

Quality Statement

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

The results presented, and comments offered refer to the sample of brickwork mortar received in CMC's laboratory on the 21st January 2021, from Nigel Copsey of the Earth, Stone and lime Company, identified as Masonry Mortar from below the Eaves on the 19th century brick building at 60 Goodramgate, York.

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For CMC Ltd.

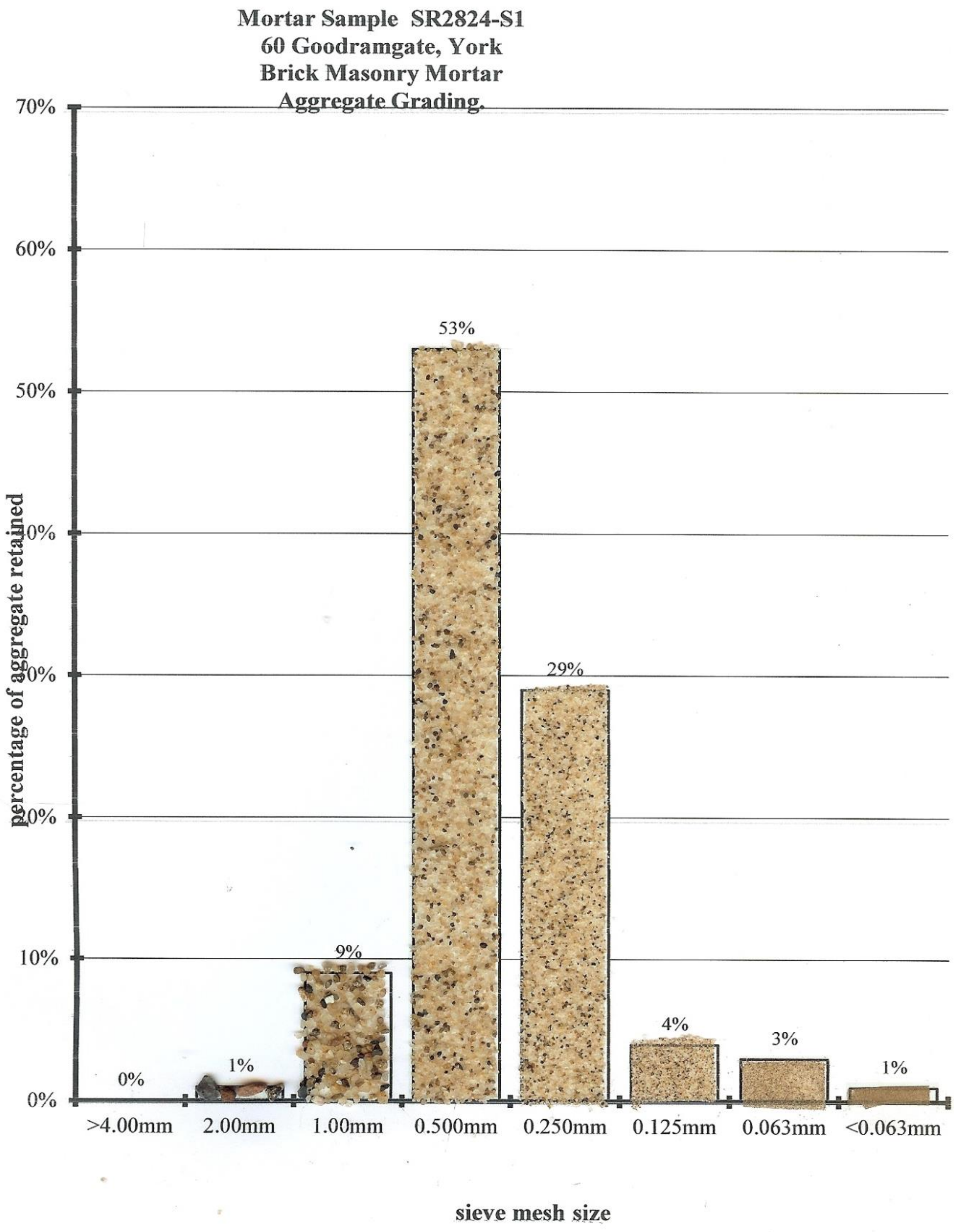

Figure No. 1: Sample SR2824-S1 – Grading of aggregate recovered from the acid digestion.

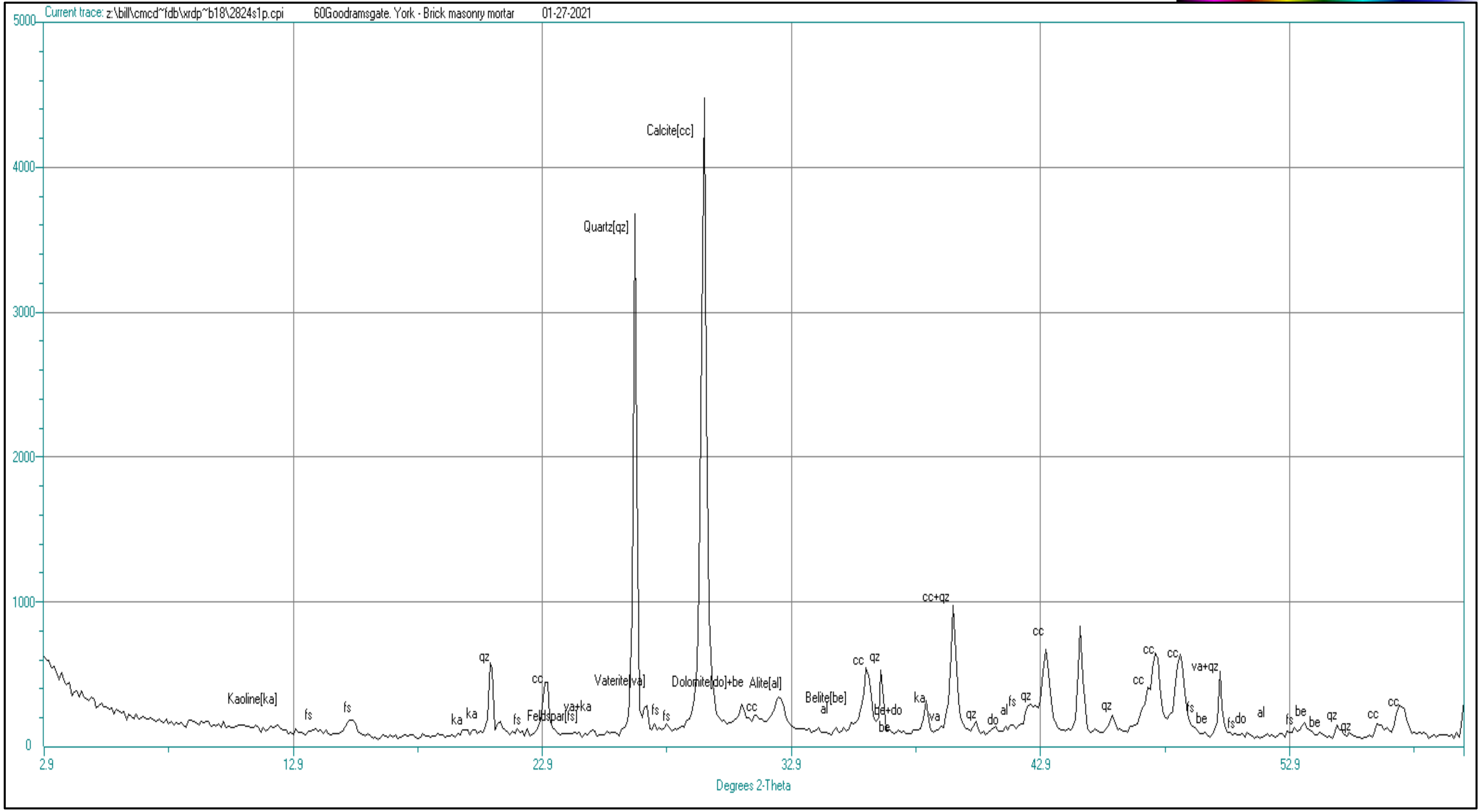

Figure No. 2: Sample SR2824-S1 – Binder rich fines from the bedding mortar sample.