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Our Ref: M/2097/21/C1
Your Ref.: Old St. Margaret's

2nd February 2021

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

Project Reference	:	Old St. Margaret's, Harwood Dale, North Yorkshire
Sample Source	:	Mid 17th Century ruined church building
Sample Description	:	Masonry mortar
Date Received	:	21st January 2021
CMC Sample Ref	:	SR 2823 - S1
Method of Test	:	Mix composition by acid digestion, with binder type by XRD analysis following in-house procedures. With the fabric condition assessed from a thin section examination.

Sample

A sample of wall core mortar submitted by Nigel Copsey of the Earth, Stone & Lime Company, was received. in CMC's Stirling laboratory on the 21st January 2021, with the sample identified as masonry mortar from the ruin of Old St. Margaret's Church, Harwood Dale, North Yorkshire.

Information relating to the sample was received by email on the 14th January 2021, with confirmation that the sample was despatched to CMC received on the 20th January 2021, along with the instruction that it was to be submitted to analysis to establish the composition of the mortar used in the masonry, its condition, and any other useful information that may be gleaned from the examination and analysis.

On receipt in the laboratory, the sample details were entered into the sample register and the unique sample identification number SR2823 allocated.

The details of the sample received is presented below:

CMC Ref.	Client Ref	Location Sampled
SR2823 - S1	Mortar	Mortar sampled from the masonry of the ruined Old St. Margaret's Church, Harwood Dale, North Yorkshire.

Method of Test

Prior to preparing the sample for analysis the sample was photographed on receipt, with its mass and size recorded. The sample was then submitted to an examination with the aid of a stereo-binocular microscope at a magnification up to x10, during which the sample was exposed to a series of *ad hoc* droplet tests employing a range of reagents and indicator solutions. This was 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 sub-sample was prepared for analysis by X-ray Diffraction (XRD). The sub-sample consisted of the core of several inclusions picked from the mortar, with a little of their encapsulating paste. The sample was prepared by gently grinding it in an agate mortar and pestle to separate the binder from any aggregates that may be present from the adhering paste, with a binder rich sub-sample recovered by sieving the disaggregated material over a 63µm sieve.

The powdered sample was backpacked into a proprietary sample holder in preparation for presentation in the diffractometer. With the sample 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°.

Following the initial examination, a further representative sub-sample sample was prepared from the whole mortar sample, with this dried to a constant weight and the material lightly ground in a mortar and pestle in preparation for digestion of the binder in dilute hydrochloric acid. The acid digestion was carried out following standard procedures which were modified to permit a more dilute acid to be employed, along with shortened dwell period in the acid, as it was indicated that the aggregate may contain limestone.

On completion of the acid digestion the aggregates were recovered by vacuum filtration, washed to remove any residual acid, dried and graded through a nest of British Standard sieves.

In addition to the above a slice was cut from an intact piece of mortar and used in the preparation of a petrographic thin section. This was used to assist in establishing the form in which the binder was used in the production of the mortar and to permit correction of the mix composition for any acid soluble aggregate and lime inclusions that were observed in 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
SR2823-S1	Mortar	130.6	56.2 x 40.9 x 31.0	14 + fines	2.5Y 6/2 "Light brownish grey"

The sample received consisted of fourteen intact pieces of mortar along with a quantity of fine fragments. The mortar was firm and moderately hard and well compacted, and required firm persistent finger pressure to break, though once disrupted the mortar could be powdered under firm finger pressure.

The received sample was moist and therefore a sub-sample was dried to a constant weight and its as-received moisture content determined, and found to be 18.9% by dry mass.

It was observed that all of the intact pieces contained an abundance of lime inclusions, with the inclusions noted to range from sub-angular to sub-round in shape with a minor proportion noted to be irregular to flaky. The largest inclusion was measured at 8.2mm, but most were finer than 3.5mm in size.

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

Water droplet tests were carried out, which involved placing droplets from a pipette onto both the outer mortar surfaces and onto freshly fractured surfaces. The test confirmed that the mortar pieces all contained a well-connected pore structure, with the droplets, placed absorbed very quickly and diffused through the mortar to depth.

Plates No. 1 & 2: The left plate above shows the mortar sample as received, in which the abundance of lime inclusions can be clearly seen. The right plate shows the intact pieces selected for analysis, with the larger fragment used in the preparation of a thin section.

Plates No. 3 & 4: The left plate is a close-up of the largest fragment showing the abundance and distribution of the lime inclusions. The right plate shows a view of a freshly cut surface through the thickness of one of the fragments. Again, note the abundance and variation in size and shape of the lime inclusions, white, in the above images. Also note the presence of a wood fragment, arrowed in the right plate.

Results of XRD Analysis

To confirm if the binder was a lime, and determine if it was hydraulic, or not, a number of the lime inclusions were collected and prepared for analysis by X-ray Diffraction (XRD). This would assist in confirming if any hydraulic components were present in the lime used in mortar production.

The result is presented in the following figure, in the form of a labelled X-ray Diffractogram:

Figure No. 2: Sample SR2823-S1 – Combined Lime inclusions picked from intact mortar pieces.

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

- **cc** = Calcite ($CaCO_3$) calcium carbonate, carbonated lime binder, and also present in any limestone present in the aggregate,
- **be** = Belite (C_2SiO_4) *di*-Calcium silicate, hydraulic clinker component in lime & cement,
- **qz** = Quartz (SiO_2) dominant aggregate component in the sand, with trace proportion within the lime inclusions,
- **fs** = Feldspar, mostly of Anorthite of the Plagioclase group, along with Sanidine, a Potassium feldspar, present in the sand and also in trace proportions within some of the lime inclusions,
- **di** = Dickite, clay of the Kaolinite group of minerals, present as a component of the aggregate, within some lime inclusions and/or as a component of any adhering soiling.

On the basis of the XRD analysis, it is indicated that mortar sample was made from a lime binder, which displays the characteristics of a non-hydraulic, high calcium, air lime, However, it is also indicated that some of the limestone used was possibly a "dirty limestone" containing the components that could, and, in the sample analysed, did form a trace proportion of a hydraulic component, confirming that the temperature reached in the kiln was likely to have exceeded 850˚C. The component detected, in trace proportions, is Belite (C_2S) although the proportion is unlikely, in the sample analysed, sufficient to impart a notable hydraulic property to the mortar.

The mortar is fully carbonated and there was no evidence of the mortar having been affected by reactions with any form of environmental sulphate, or other deleterious contaminants.

To provide additional information the data from the XRD analysis was processed by Rietveld Refinement, in the Maud computer program, to permitted quantification of the minerals and crystalline material present. The results obtained are presented below:

Sample Ref.	**SR2823-S1**
Material	**Lime Inclusion**
Component	**Proportion (% by Mass)**
Calcite ($CaCO_3$)	93.7
Belite (Ca_2Si_4)	0.5
Quartz (SiO_2)	3.3
Feldspar (Anorthite)	0.9
Feldspar (Sanidine)	0.1
Dickite (Clay)	1.5
Total	**100.0**

The binder is composed of calcite (Carbonated lime), along with a minor proportion of quartz, feldspar and clay, which is not uncommon, as many limestones also contain a low proportion of other rock minerals within their fabric. However, a proportion of the trace minerals detected may be present as contaminants from the aggregate, as well as components of the quicklime lime used as the binder.

Mix Composition

To permit the recovery of the aggregate for grading analysis, and to ascertain the mortar mix composition a further representative sample was prepared and submitted to determination of mix composition by acid digestion. As it was indicated that there was a possibility that some limestone was present, the strength of the acid was reduced (5%) rather than the usual 10% solution.

The dwell was also time kept to a minimum, just sufficient to dissolve the paste and the inclusions. To achieve this, it was necessary to repeat the digestion twice, to ensure that all of the lime, including inclusions, were consumed.

The result of the composition analysis carried out, corrected for limestone content is presented below:

Sample Ref. No.	**SR 2823-S1**	
Binder type (from XRD)	Non-Hydraulic Lime	
	Quicklime	Lime Putty
Lime: Aggregate Ratio	1.0 : 3.1	
Weight proportions calculated mix ratio by dry mass.		
Lime	1.0	1.0
Aggregate	5.5	2.5
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	1.55	1.7

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

Sample Reference	**SR2823 – S1 Masonry Mortar**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100
4.00mm	1.7	98.3
2.00mm	4.4	93.9
1.00mm	0.9	93.0
0.500mm	11.8	81.2
0.250mm	28.0	53.2
0.125mm	29.3	23.9
0.063mm	11.6	12.3
Passing	12.3	

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

The aggregate particles are dominated by quartz grains, with Oolitic Limestone and sandstone/siltstone fragments, with altered lithic grains, quartzite, feldspar and clay minerals. The particles are sub-angular to sub-round in shape.

The high proportion passing the 63 micron sieve was found on examination and XRD analysis to be a mixture of very fine quartz grains, feldspar and clay minerals, and it is likely that the aggregates were used in the as-dug condition.

Microscopic Examination

To clarify the form in which the binder was used and permit comment on the mortar fabric a petrographic thin section was prepared from an intact piece of the mortar

Aggregate

The aggregates in this mortar sample are dominated by quartz grains with limestone, sandstone, and a low proportion of altered lithic fragments present, mostly of quartzite, chert and ferruginous sandstone, along with opaque minerals and a trace proportion of ash clinker and pieces of wood. It is not possible to clearly identify all of the opaque minerals by optical microscopy alone, although ironstone, coal and charcoal are all considered to be present. The coal and the charcoal possibly carried over with the lime from the kiln, as many show altered, burnt, margins.

The aggregate grains, are typically sub-angular to sub-round in shape, and locally flaky, with many displaying water worn margins, with the shape and the texture of the aggregates suggesting a glaciofluvial source for the sand.

The sand grains range from 0.04mm to 4.6mm and are dominated by quartz grains, limestone fragments, sandstone/siltstone particles, along with minor quartzite, and chert, with rare altered igneous rock types, ironstone, coal, wood and ash clinker. The aggregate would be classed as a medium to fine grained sand, with a proportion of silt and clay sized material (<0.63µm) diffused throughout, which may infer that the aggregate was used as an as-dug material rather than as a processed (washed) sand.

Binder

The paste has the appearance of having been produced from a non-hydraulic lime, with a high proportion of sub-angular to sub-round inclusions, which also, locally, appear as irregular and rounded features. The more rounded grains retaining a partial rock imprint inferring an Oolitic limestone as the origin of the lime.

Lime inclusions are variably slaked, with rare fragments observed to retain residual rock fabric, which would infer that calcining of the limestone was, in part, incomplete, along with the shape and texture of others inferring that they had been locally poorly slaked at the time of mixing.

A proportion of the inclusions display sharp outer margins, some of which also appear to be incompletely slaked, whilst others display both under burnt and over burnt outer margins. This is not uncommon in early hot mixed mortars, where incompletely calcined lime had not been screened out. However, all of the inclusions are now fully carbonated.

The variation in shape, of the inclusions, along with the well compacted encapsulating paste both inferring that the mortar had most likely been mixed as a Hot Mixed Mortar (HMM), where the quicklime was mixed with the sand, but also placed as a hot placed mortar (HPM), i.e. whilst a proportion of the lime was still hydrating as it was placed in the structure. This would account for the abundance of lime inclusions and incompletely hydrated lime observed in the sample.

The paste is fully carbonated throughout the sample and there is evidence of localised leaching, with loss of lime from both inclusions and locally from the paste around the outer perimeter of the intact mortar fragment examined. There is, however, only little, very localised, redeposition of calcite, with this mostly observed along crack margins and lining voids from which lime inclusion had been depleted.

The lime inclusions observed ranged in size from 0.2mm to 3.1mm in the section examined, but mostly these are <2.0mm.

There is an abundance of inclusions observed within this sample that retain a patchy rock imprint which would infer that the limestone used, in the lime production, was a bioclastic limestone containing Ooliths, similar to that observed in limestone in the aggregate.

Although none of the inclusions contain any clear hydraulic components within them, rare Belite clusters can be seen within the paste adjacent to inclusions. These are small and although present, their abundance would be insufficient to classify the binder as a hydraulic lime and it is concluded that the lime used in the production of the mortar performed essentially as a non-hydraulic, high calcium lime. The presence of Belite, may be due to a low proportion of the limestone burnt, in the lime production, being contaminant from a dirty limestone horizon, or, alternatively, being present in the ash from the fuel used in the kiln.

Voids and microcracks

The voids observed in the mortar are mostly sub-angular to irregular in shape, and are mostly dissolution voids from the post placement depletion of lime inclusions, although there are also a few entrapped air voids, present as placing artefacts. The voids range in size from 0.2mm to 3.3mm in size, many of which retain Calcite rims from leached and redeposited lime.

Cracks are minor, with those present having the appearance of early drying shrinkage cracks, and as placing features, with only minor redeposited calcite linings observed within the crack paths. The crack margins are generally dense with the cracks appearing not to have acted as fluid migration channel ways. Cracks range in width from <0.01mm to 0.18mm.

Modal Analysis

The mix proportion 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, which 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.

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

Sample Ref:	SR2823-S1	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	14.6	14.6
Quartzite	2.8	2.8
Sandstone fragments	4.4	4.4
Limestone	10.1	10.1
Lithic Fragments + Feldspar	7.2	7.2
Ash & clinker	0.5	0.5
Opaque Coal + Ironstone	4.3	4.3
Lime inclusions	-	17.4
Total Aggregate	**43.9**	**61.3**
Binder (Lime)	37.0	37.0
Lime inclusions	17.4	-
Clinker	0.1	0.1
Secondary products/Calcite	1.6	1.6
Total Binder	**56.1**	**38.7**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**12.6**	**12.6**
	Total	**Effective**
Binder: Aggregate Ratio	**1.0 : 0.78**	**1.0 : 1.58**

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

Photomicrographs:

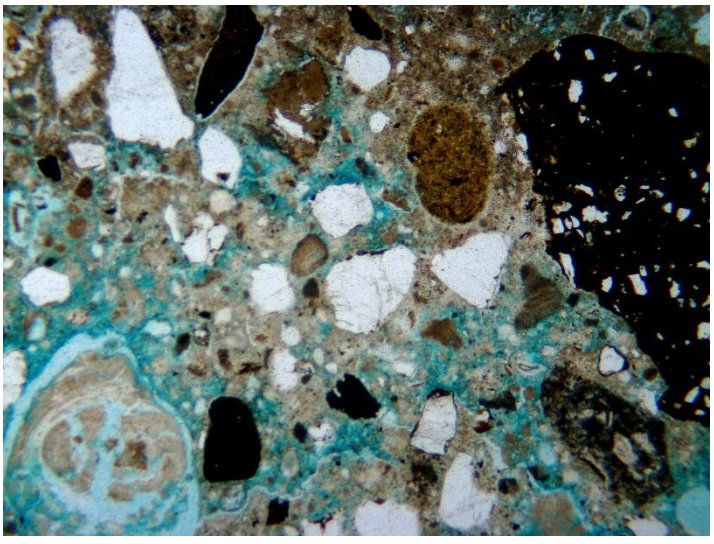

Plate No. 5:

A view in plane polarised light (ppl) of an area of the mortar, which depicts a typical area of the mortar fabric. An ironstone particle can be seen on the right side of the of the plate, with a partially disrupted Oolitic limestone particle in the lower left corner. The dense paste is fully carbonated and encapsulates a mixed suite of aggregates, dominated by quartz particles, along with siltstone, coal fragments, and altered igneous rock fragments

Porosity and voids are highlighted by the blue dyed resin.

Field of view 2.4mm.

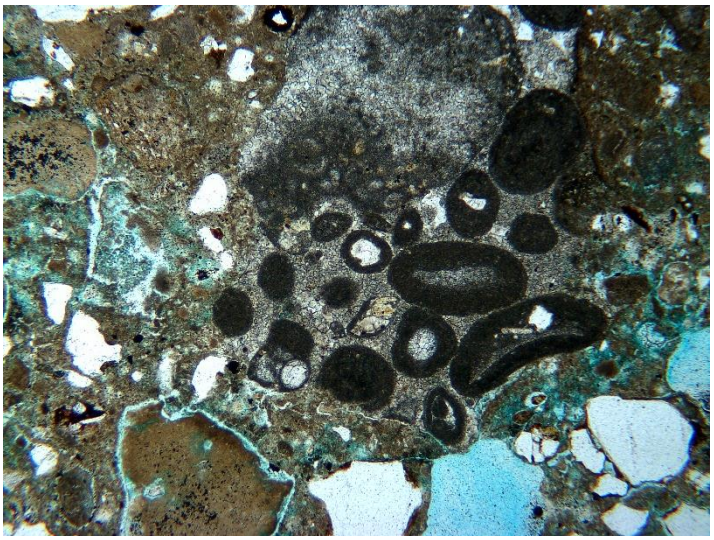

Plate No. 6:

Another view in plane polarised light (ppl) where a large limestone fragment can be seen filling centre to upper right part of the plate. The limestone is from a bioclastic rock containing Ooliths, shell fragments and foraminifera, etc. There is a calcined lime inclusion in the lower left, which has only been partially hydrated, and is surrounded by a shrinkage crack highlighting its perimeter showing that it did not form part of the paste. The aggregates in view on are dominated by quartz grains, with minor opaque minerals and ash clinker. Porosity and voids are highlighted by the blue dyed resin. Field of view 2.4mm.

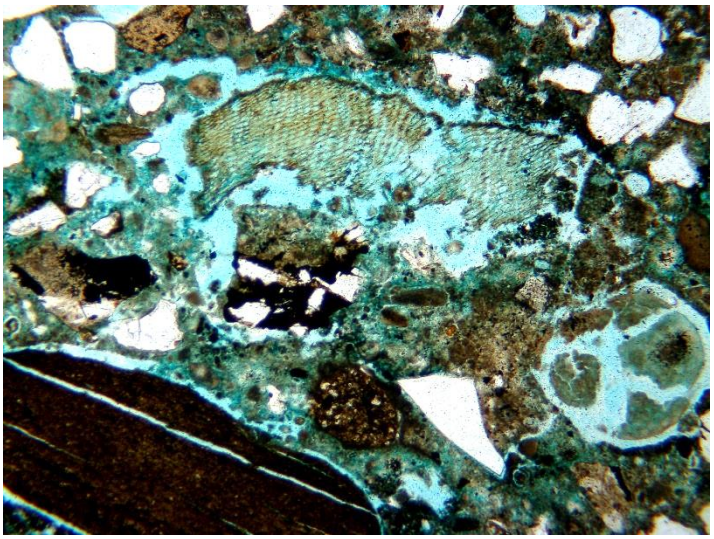

Plate No. 7:

Another view in plane polarised light, ppl, where a fire singed wood fragment can be seen in the centre of the plate. With a lime inclusion in the lower right and a large siltstone particle in the lower left. The paste within the lime mortar is fully carbonated and locally displays microporosity. The paste around the wood fragment has been depleted due to leaching. The aggregates in view are again dominated by quartz grains, mostly white in this image, with minor opaque minerals, black, feldspar and altered lithic grains. Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

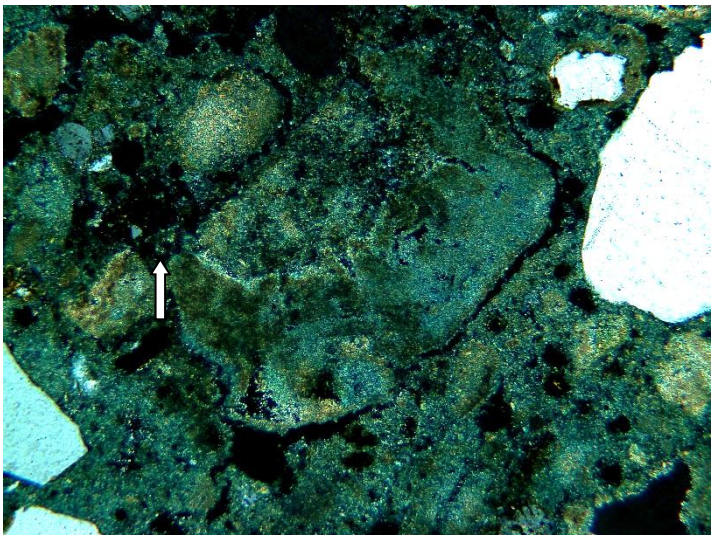

Plate No. 8:

A view in cross polarised light, xpl, showing a partially overburnt limestone particle in the centre of the plate, with the lime only partially diffusing into the paste, lower left and upper centre. The remainder of the inclusion is clearly defined by a sharp boundary. A small clinker grain can be seen in the left side of the particle (Belite), arrowed in plate. The aggregates in view are again dominated by quartz grains, white in this image, upper 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 Old St. Margaret's church, it is indicated that the mortar was made from a non-hydraulic air lime and a mixture of medium to fine natural sand. The lime having been used in the form of a quicklime. The mortar shows all the features of having been mixed as a Hot Mixed Mortar and placed whilst still slaking, as a Hot Placed Mortar.

Details of the mix used is summarized below:

Sample Ref.	**SR2823- S1**
Volume Proportion by acid digestion	
Binder content (Quicklime)	1.0
Fine Aggregate (Sand)	1.6
By modal analysis	
Binder: Agg. by vol. (Total)	1.0 : 0.8
(Effective)	1.0 : 1.6

The aggregates in the mortar are a natural quartz rich, as dug sand, containing limestone and lithic fragments.

Sample Reference	**SR2823 – S1 Masonry Mortar**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100
4.00mm	1.7	98.3
2.00mm	4.4	93.9
1.00mm	0.9	93.0
0.500mm	11.8	81.2
0.250mm	28.0	53.2
0.125mm	29.3	23.9
0.063mm	11.6	12.3
Passing	12.3	

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 mortar received in CMC Ltd.'s laboratory on the 21st January 2021, from Nigel Copsey of the Earth, Stone and Lime Company, which was identified as Masonry mortar from the ruined Old St. Margaret's church, Harewood Dale, North Yorkshire.

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For CMC Ltd.

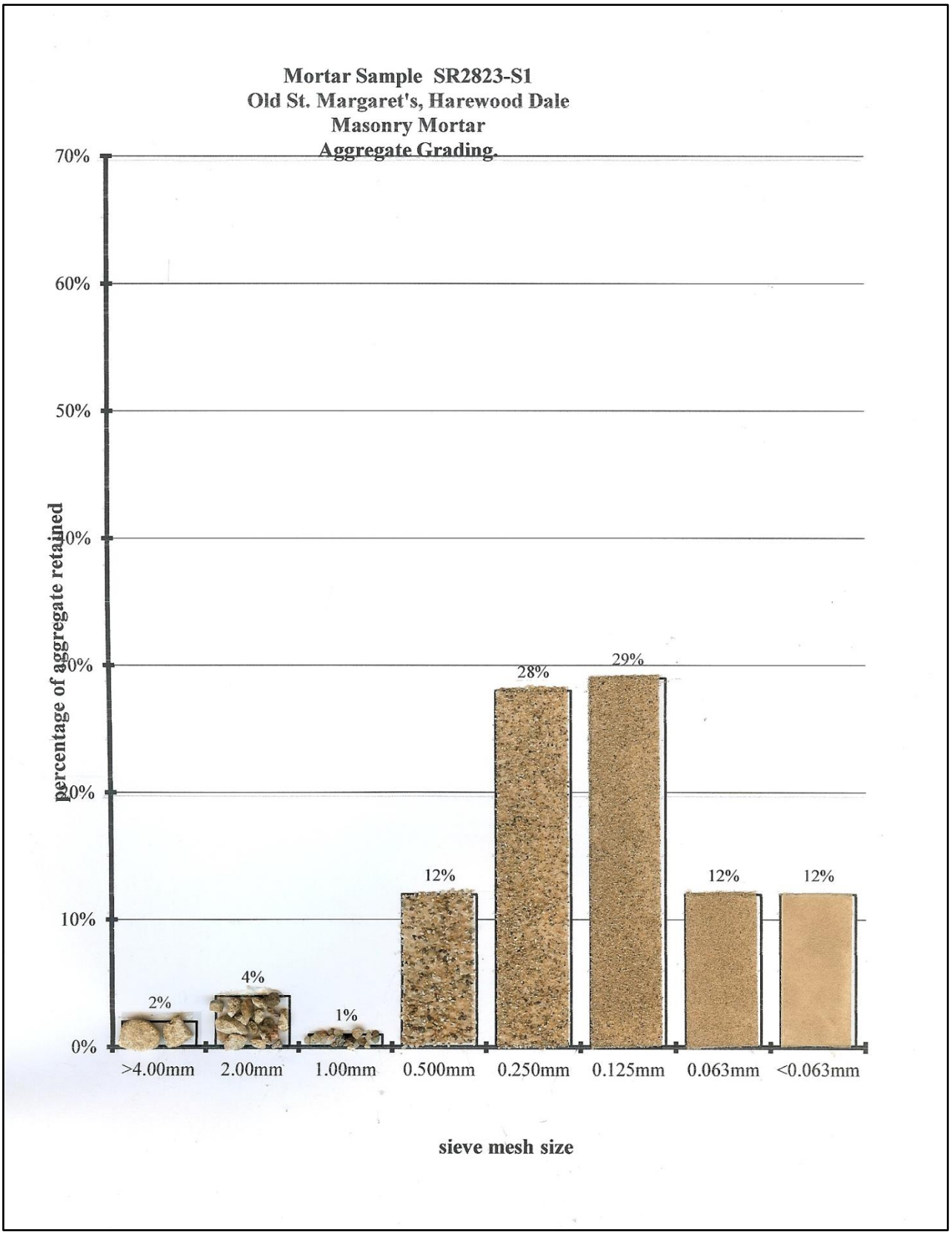

Figure No. 1: Sample SR2823-S1 – Grading of recovered aggregate following acid digestion.

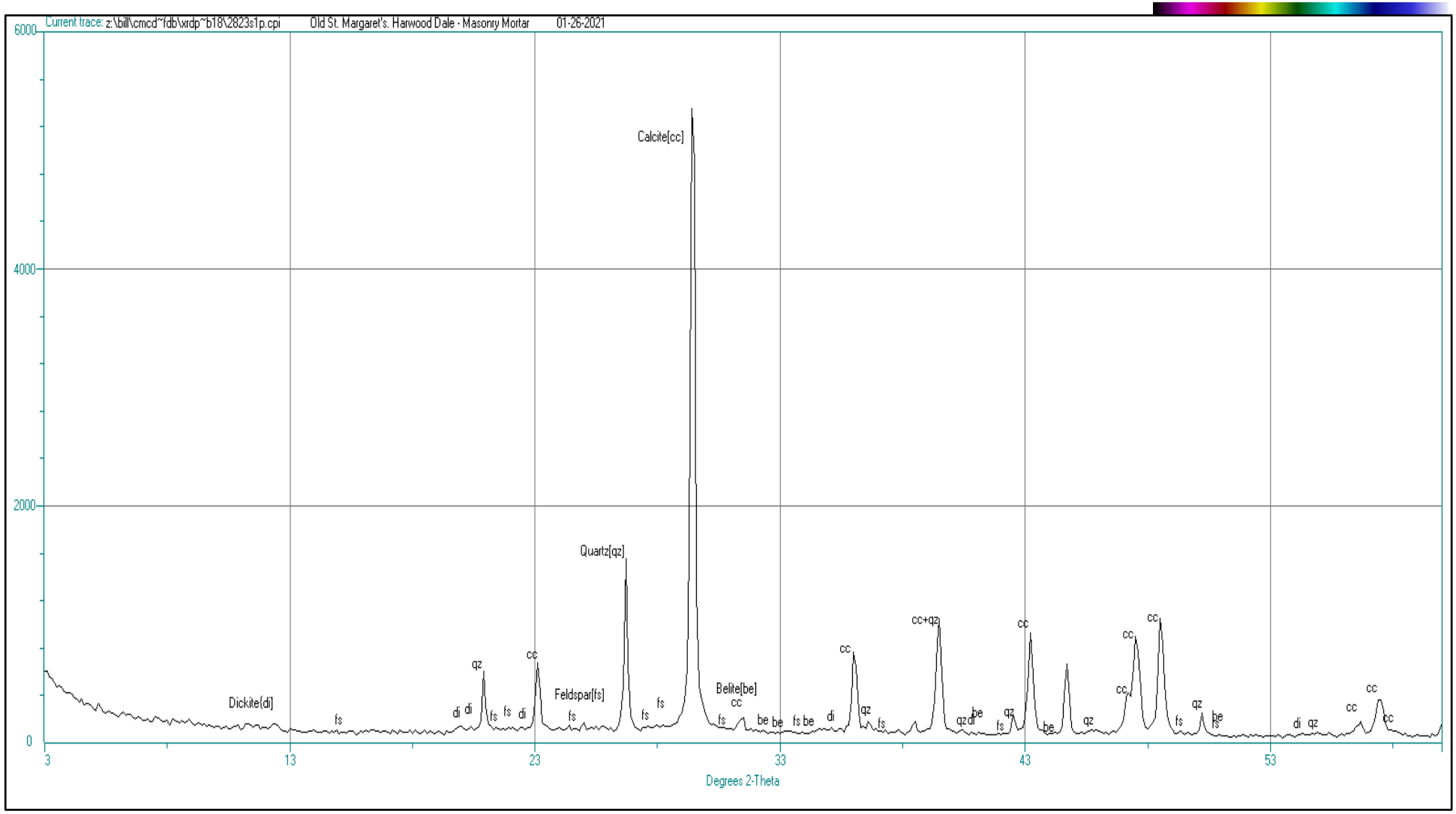

Figure No. 2: Sample SR2823-S1 – Combined Lime inclusions picked from intact mortar pieces.