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Our Ref: M/1953/18/C1
Your Re.: Stonehouse

7th January 2019

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

Project Reference	:	Stonehouse, Brook Lane, Thornton Dale, North Yorkshire
Sample Description	:	Pointing mortar to a Clay/Lime Mortared masonry Wall
Date Received	:	27th November 2018
CMC Sample Ref	:	SR 2662 – S1
Date Analysed	:	3rd 20th & 27th December 2018 and 3rd January 2019
Method of Test	:	Binder type and presence of reaction products by X-Ray Diffraction, with Mix Composition by Modal Analysis and examination of a Petrographic thin section following standard procedures.

Sample

A sample of mortar identified as "Pointing Mortar" was received in CMC's Stirling Laboratory on the 27th November 2018. The sample was submitted 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 confirming whether the mortar contained any aggregate, and if so, confirm type and quantity. The presence of other components or reaction products were also to be determined.

To achieve this, the sample was initially examined with the aid of a stereo-binocular microscope at magnifications up to x20. Following this a selection of the mortar fragments were used in the preparation of a petrographic thin section. With another sub-sample prepared and analysed by X-ray Diffraction, which would provide additional information on the type of binder employed in the production of the mortar, and, if possible, 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 SR2662-S1 allocated.

The sample details are presented below:

CMC Sample Ref.	Client Ref	Location Sampled
SR2662 – S1	Pointing Mortar	Pointing Mortar from a Clay/Lime mortared Masonry wall

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 petrographic thin section was prepared to permit the form in which the binder was made and used to be assessed, and if possible, determine the mix proportions, if aggregates are present. Other features observed were also to be recorded, as these may assist in its replication for use in conservation, restoration or repair works

Confirmation of the binder type used in the production of the mortar was determined by X-ray Diffraction (XRD) analysis. This was achieved by crushing and grinding another 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 a proprietary sample holder in preparation for presentation in the X-Ray Diffractometer.

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
SR2662-S1	Pointing	73.13	57.12 x 37.2 x 29.3	"White"

The sample received consisted of 12 small pieces of a well bound lime mortar along with a small quantity of loose fines.

The mortar was well compacted and moderately hard although the intact pieces could be broken under moderate to firm finger pressure and once disrupted the mortar could be powdered under moderate finger pressure. The mortar, although well compacted, was noted to contain a few irregular shaped voids, which appeared to be placing features with soiling of the surfaces exposed within the voids inferring that water percolation through the pointing had occurred locally.

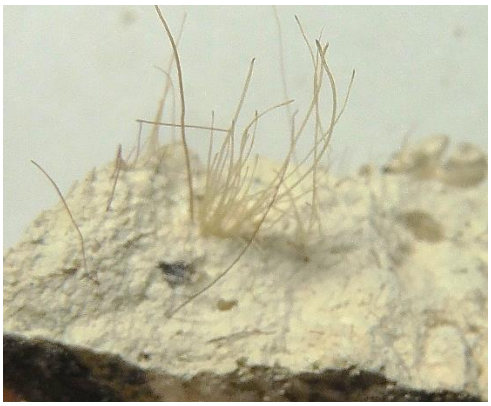
Small spherical entrained air voids were apparent, in abundance, with these distributed throughout the paste, which is typical of that observed in a lime putty mortar mix, that has been well mixed.

Small dark indeterminate 'aggregates; were observed, however, these were small, typically less than 0.2mm and these were poorly distributed and scarce in occurrence. Other aggregates observed were up to 0.76mm in size and had the appearance of quartz grains, with their abundance low and the grains randomly distributed.

It was noted that the mortar was light in weight, with this found, on measurement of the density of several fragments, to be in the region of 1330kg/m^3. This is light for an aggregate filled mortar and would tend to infer that the aggregate content is likely to be low, or of a low density form.

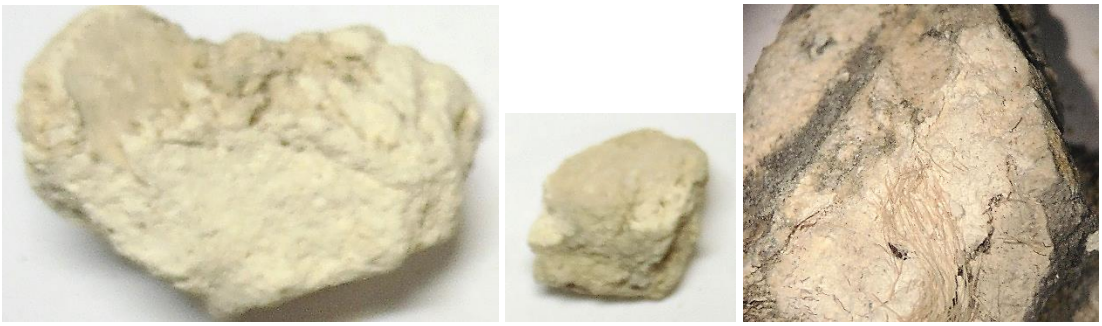
Plate No. 1: The above plate shows the intact pieces of mortar as received, which was found to consist of twelve mortar fragments and a small quantity of fines.

Plates No. 2 & 3: The left plate shows a close-up of a soiled surface within a void in the mortar, where water percolation with the deposition of both transported debris and redeposited leached lime were both apparent. The right plate shows a freshly fractured surface through the thickness of the pointing, with "aggregates" apparent along with lime inclusions and hair fibres. Tests on the fractured surfaces with a phenolphthalein indicator solution confirmed that the mortar was fully carbonated.



Plates No. 4 & 5: The above plates show fractured surfaces in which an abundance of fibre can be seen, with hair fibre occurring as clumps, poorly separated fibres, and as individual hair fibres. The hair ranged in colour from white, light and dark brown to red, with the fibres being of animal hair, possibly horse body hair.

White inclusions, possibly lime inclusions, were noted within the mortar, but these were random in occurrence, with that observed forming inclusions ranging from 0.24mm to 14.2mm in size.



Plates No. 6, 7 & 8: The above plates show close-up views of examples of inclusions within the sample. The left plate shows what appears to be a large angular inclusion (lower part of fragment). The centre plate is of a mortar fragment containing two angular inclusions, shown white in plate. With the right plate taken under the stereo microscope of a large irregular to sub-rounded putty lime inclusion, with hair fibre both at its margin and embedded into its surface.

Ad-hoc tests for the presence of organic components or the presence of linseed oil, or other waterproofing aid (Tallow, etc) all proved negative, therefore, it was concluded that the mortar did not contain any water proofers or other, non-mineral, additives.

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 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 result of the analysis, by X-ray Diffraction, is presented in the following attached Figure at the end of this report, which is in the form of a labelled X-ray Diffractogram:

Figure No. **1**: Sample SR2662-S1, Powdered sample of Pointing mortar, ex the Stonehouse.

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,
- **fr** = Friedel's Salt ($Ca_4Al_2O_6Cl_2 10H_2O$) Calcium Aluminium Oxide Chloride Hydrate, Hydration product, possibly from a minor pozzolanic reaction with ash in feed stock,
- **gy** = Gypsum ($CaSO_4 2H_2O$) Calcium Sulphate Hydrate, reaction product between lime in binder and environmental sulphates.

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

Component	Proportion (% by Mass)
Sample:	**Mortar sample**
Calcite	96.0
Quartz	2.1
Friedel's Salt	1.0
Gypsum	0.9
Total	100.0

Based on the XRD analysis, it is indicated that the mortar is essentially a high calcium lime mortar mix, with a trace proportion of quartz. The latter is likely to have been present as a contaminant in the feedstock rather than a purposefully added component.

Minor reaction products were detected in the sample analysed, with two forms detected, Friedel's Salt which is a hydration reaction product, which may infer a very minor proportion of pozzolanic material in the ash, or the presence of potentially hydraulic component from within some of the overburnt lime inclusions, such as aluminates in the limestone, i.e. from feldspar or clay minerals. The second reaction product is in the form of Gypsum, and this is likely to be indicative of a post placement reaction with environmental sulphates, from contaminants rather than infer that a low proportion of gypsum had been added to the mortar at time of mixing.

Microscopic Examination

To further clarify the form in which the binder was made and used a petrographic thin section was prepared from several fragments of intact pieces of the mortar. These were cast in, and impregnated with, a blue dyed epoxy resin. One side of the cast block was cut and polished prior to being mounted on a glass slide (50mm x 75mm), with the sample aligned to give the maximum area of the mortar fragments 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.

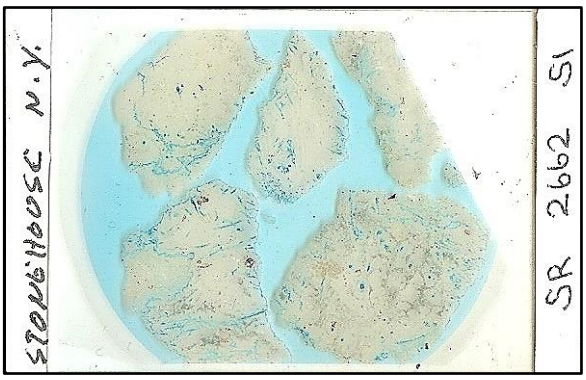


Plate No. 9:

Thin section prepared for microscopic examination and Modal Analysis.

Aggregate

The aggregates in the mortar sample are dominated by calcareous limestone fragments, with Dolomite and minor quartz grains with other heavily altered lithic fragments also present.

The limestone aggregate grains are predominantly sub-angular to sub-rounded, and with a majority of the grains displaying partially abraded and rounded margins. This may infer that the aggregates were from a crushed and ground limestone, i.e. limestone dust. The aggregate grains range in size from 0.012mm to 1.9mm (medium silt to coarse sand grains in size).

Limestone is present as fresh angular fragments of both Oolitic and micritic limestone, along with a low proportion of Dolomitic limestone fragments, with no evidence of having been calcined. However, there is also an abundance of other Limestone fragments present, which consist of overburnt or partially burnt fragments. This, along with partially slaked limestone fragments suggesting that the mortar appeared to have been mixed from quicklime or an unscreened slaked lime putty, which included both under burnt and overburnt limestone fragments, and limestone dust as an aggregate.

A low proportion of ash material, presumably from the kiln is also present, but there is only very miner indications that any of this had acted as a pozzolan, and not sufficiently, with regard to quantity, or reactively, to have impacted on the properties of the mortar.

Minor opaque minerals were also present, and these include residue from the kiln fuel, which are irregular in shape, some are granular in texture, though none are typical of coal or coal ash residue. Others opaque grains may be iron rich minerals present in the feedstock or aggregate.

A proportion of animal hair was observed within the mortar, with the hair being typical of that obtained from horse body hair, though this would require further analysis to confirm. The hair is in sufficient proportion to infer that it was purposely added as a reinforcement.

Binder

The binder is a non- hydraulic High Calcium Lime, with the limestone calcined appearing to be a mixture of Oolitic limestone and micritic limestone, possibly from the same source. The calcining appears to have been at the lower range of the temperature scale, as there is was a high proportion of partially burnt inclusions observed. Although, a very low proportion of overburnt fragments are also present, these are low in abundance, and probably are associated with localised hot-spots in the charge, which is not an uncommon occurrence in early lime kilns.

The binder, and some of the inclusions have the appearance of a putty, whilst a low proportion of the inclusions show the presence of partially slaked material, including partially burnt/slaked limestone fragments. This may infer that the quicklime was slaked and run to a putty before use, however, if so, it was not screened prior to use. Alternatively, and probably more likely, the mix was prepared from slaking the quicklime along with the limestone dust and the whole mixed wet, initially to a workable consistency, but used cold after remixing, with the addition of hair fibre, to minimise shrinkage cracking.

There is limited evidence of water percolation, both along placing artefacts and fine shrinkage cracks acting as channel ways, with very localised lime leaching, particularly at hair clump margins and at crack margins. There is very limited evidence of redeposited lime in the sample examined, although fine fringes of coarse redeposited calcite and fine gypsum crystals was observed on the walls of crack paths that had acted as channel ways for percolating waters.

Voids and microcracks

Voids and cracks are present, but these are minor in occurrence within the intact pieces examined. These appear to have formed as placing artefacts, and in response to drying shrinkage.

Voids range from 0.12mm to 0.8mm in size, but are typically <0.4mm, and mostly free of linings. However, some peripheral voids contain fine linings of secondary minerals, whereas fully encapsulated voids are generally free of coatings.

Cracks range from <0.01mm to 0.36mm in width and from 6.2mm in length to the full width of the fragment in which they occur. Fine cracks, <0.03mm are generally free of linings, whereas, several of the wider cracks have acted as fluid migration channel ways and these show areas of depleted paste at their margins, with localised redeposition of calcite, and locally of gypsum, at their throats.

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

Sample Ref:	SR2662-S1	
	% By Volume	
Component	Inclusions as Binder	Inclusions as Aggregate
Quartz	1.4	1.4
Lithic fragments	1.0	1.0
Limestone (Calcareous)	23.9	23.9
Limestone (Dolomitic)	4.5	4.5
Opaque	2.4	2.4
Ash	0.6	0.6
Inclusions	-	9.2
Total Aggregate	**33.8**	**43.0**
Binder (Lime)	55.4	55.4
Lime Inclusions	9.2	-
Secondary Minerals, Calcite	1.3	1.3
Secondary Minerals, Gypsum	0.3	0.3
Total Binder	**66.2**	**57.0**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**3.2**	**3.2**
Hair	**4.0**	**4.0**
Binder: Aggregate Ratio	**1.0 : 0.51**	**1.0 : 0.75**
Lime: Agg :Hair % by Vol	**64 : 32 : 4**	**55 : 41 : 4.0**

Table No. 1: Result of modal analysis (900-point count) on the thin section

Photomicrographs:

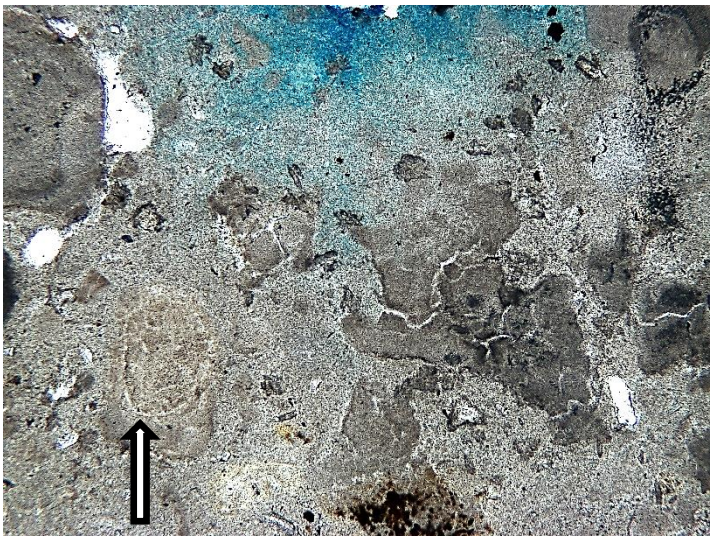


Plate No. 10:

A view in plane polarised light (ppl) showing a typical area of the mortar, which has a relatively dense fabric. Within the paste there are fine angular fragments of calcined and partially hydrated lime inclusion, centre and lower right, note cracks within these darker fragments.

A fully slaked, but confined, putty inclusion can be seen in the lower left, arrowed. With a partially burnt, and unslaked, limestone fragment in the upper left corner.

Porosity is highlighted by the blue dyed

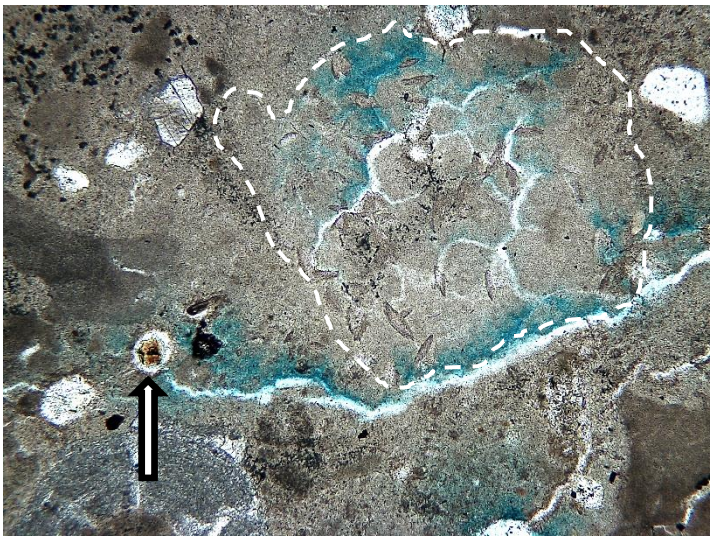


Plate No. 11:

Another view of an area of the mortar, again in ppl.

In this view there is a large sub-rounded putty inclusion, centre and upper right, in which the lime is fully slaked, and displays the characteristic shrinkage cracking of a putty, its outer margin is highlighted by a broken line. An unburnt Oolith can be seen in the lower left, with minor fine quartz grains and a hair fibre cut perpendicular to the plane of view, arrowed

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

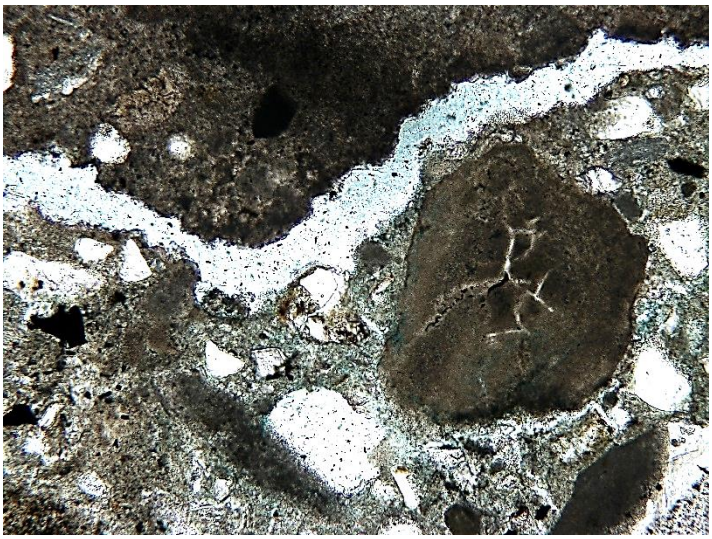


Plate No. 12:

Another view in ppl, of an area with a channel way transecting the fragment. The paste in the upper part is dense and contains clusters of ash, whilst the paste in the lower part is locally micro porous. An overburnt limestone particle can be seen in the centre right, this has not slaked and is bounded by a fine shrinkage crack, this acts as an aggregate particle in the mix. Note the abundance of sub-angular quartz grains (white) in the lower part of the plate

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

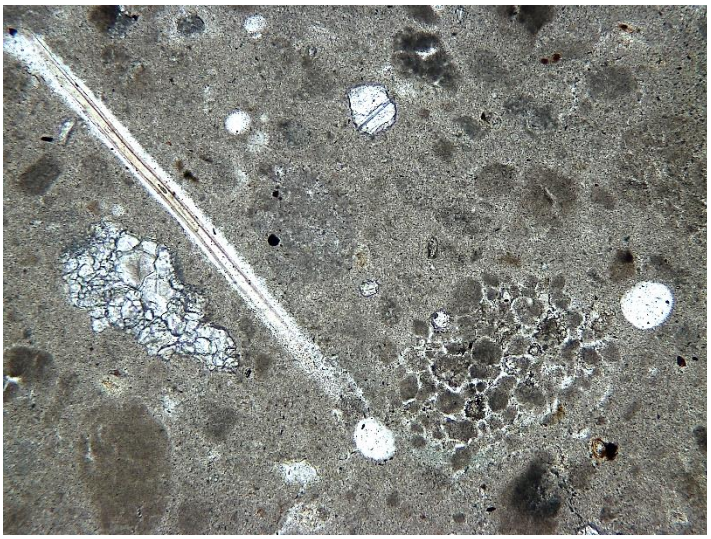


Plate No. 13:

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

The paste is relatively dense and contains an abundance of both small lime inclusions and limestone dust fragments. An unburnt dolomite fragment can be seen in the lower left along with an overburnt and partially hydrated limestone fragment in the lower right, with small fully slaked inclusion in the centre and upper right.

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 pointing to the clay mortared wall at Stonehouse, Brook Lane, Thornton Dale, North Yorkshire, it is indicated that the mortar is likely to be original, with the quicklime produced by the burning of local limestone.

The lime appears to have been moderately well slaked, and although the quicklime appears to have contained a proportion of over and under burnt fragments most of the quicklime had slaked to putty in the mix. However, given that the lime was mixed with a limestone dust, it is indicated from the thin section that the quicklime was mixed with the dust prior to slaking, with the whole slaked wet and probably remixed prior to use in the form of a stiff plastic mix applied cold.

A proportion of hair reinforcement had been added to the mortar before use, and given the good condition of the hair, it is unlikely that it would have been added to a "hot" mortar, again indicating that the mortar was applied cold.

There was no evidence to suggest that the mortar had contained any linseed oil or other organic components to aid water repellence.

Details of the mix used is summarized below:

Sample Ref.	**SR2662-S1**
Volume Proportion by	***Modal Analysis***
Binder content (Lime)	1.0
Fine Aggregate (Limestone Dust)	0.75
Hair Fibre	0.07

The aggregates in the masonry mortar is a fine crushed limestone dust, typically <2.0mm in size. The limestone containing, Oolitic, Micritic and Dolomitic types, with minor fine quartz grains, along with a trace proportion of other weathered lithic particles. The latter most likely present as impurities within the limestone.

A low abundance of ash was apparent in the mix, but no clinker and, therefore, it could not be confirmed whether the kiln had been fired with coal or wood, nonetheless, however, given the low occurrence of overburnt limestone particles, and the high proportion of under burnt particles, which along with the absence of clinker, would suggest that the limestone was most probably calcined at the lower temperature range and, therefore, wood was perhaps the most likely fuel used in the burning process.

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 lime pointing mortar received in CMC's laboratory on the 27th November 2018 from Nigel Copsey of the Earth, Stone & lime Company, which was identified as pointing from a clay mortared masonry wall in the Stonehouse, Brook Lane, Thornton Dale, North Yorkshire.

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For CMC Ltd.

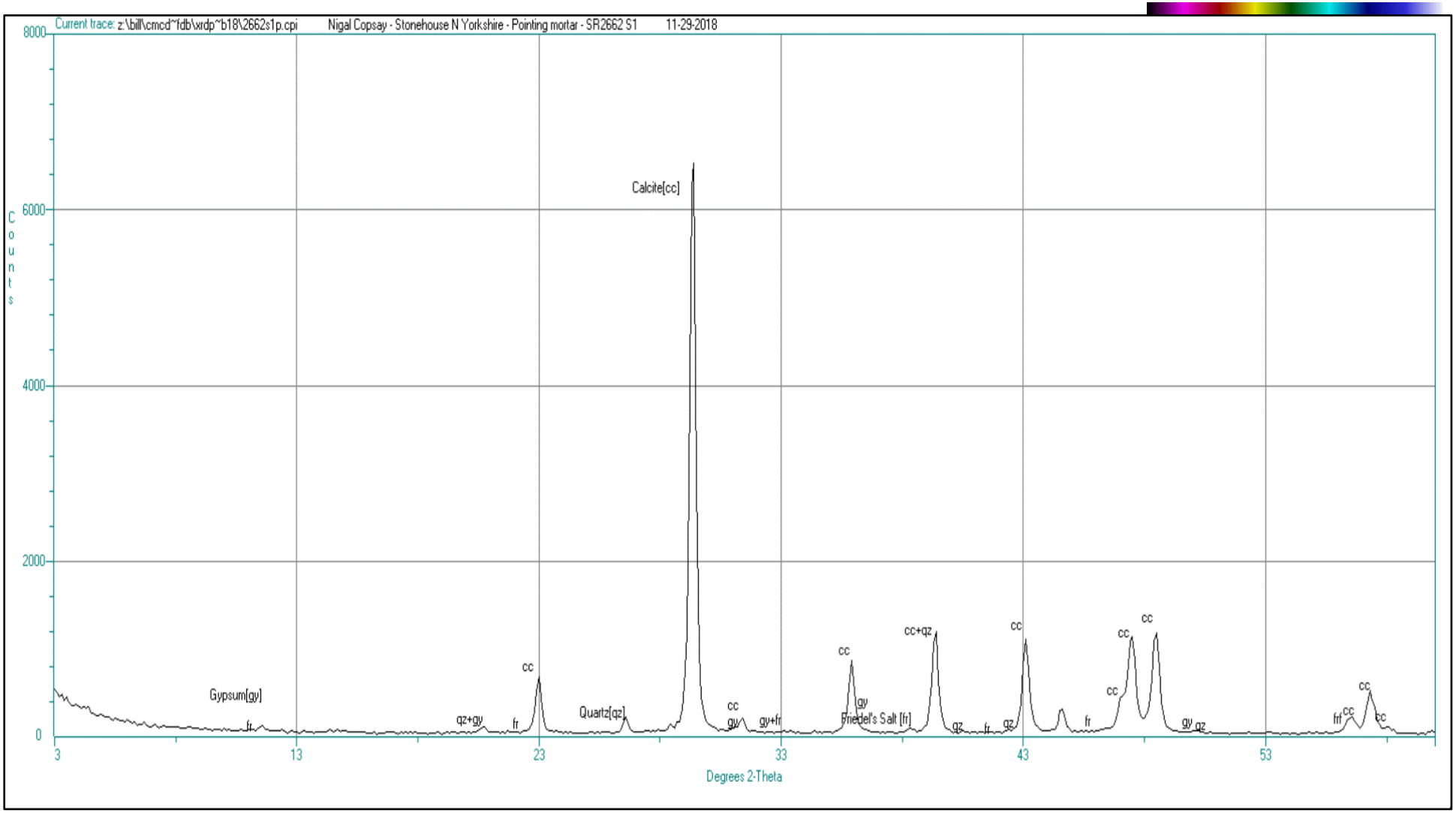

Figure No. 1: Sample SR2662-S1, Powdered sample of Pointing mortar, ex the Stonehouse, Thornton Dale.