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Report on the Examination and Analysis of Samples from 15th & 19th Century Mortars

York Minster, York

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Table of Contents

1.0 Introduction 3

2.0 Sample 3

3.0 Methods of Examination and Analysis 4

4.0 Macroscopic Examination 4

4.1 Macroscopic examination of samples 5

5.0 Microscopic Examination 10

5.1 Sample SR2600-S1 – Nosterfield washed river sand from the river Ure 11

5.2 Sample SR2600-S2 – Sand from the river Ouse, York 13

5.3 Sample SR2600-S3 – 15th Century Mortar, from York Minster 15

5.4 Sample SR2600-S4 – 19th Century Mortar, from York Minster 18

5.5 Sample SR2600-S5 – Highmoor Limestone, proposed for Quicklime production 22

6.0 Analysis by X-Ray Diffraction 24

7.0 Mix Composition Analysis 26

7.1 Mix Composition Analysis by Acid Digestion 26

8.0 Summary 27

9.0 Quality Statement 29

Aggregate Filled Histograms Figures No. 1 to 4

X-Ray Diffrctograms Figures No. 5 to 12

Trial Lime Burn and Mortar made with quicklime from Highmoor Limestone Appendix "A"

1.0 Introduction

Further to receipt of a series of telephone calls and emails between CMC Ltd and Laura Cotter, Research and Development Project Manager for the York Minster Fund, over the period 15th to 23rd May 2018, a batch of samples was received in CMC's Stirling laboratory on the 29th May 2018.

The samples were identified as 15th and 19th Century mortars, two sand samples from the local rivers, along with a piece of limestone, being considered for the production of quicklime, to be used in mortars proposed restoration works

Instruction to carry out the examination and analysis of the samples, as per the proposed laboratory programme, submitted by email on the 18th May 2018, was received by email, from Laura Cotter BEng MIET, on the 23rd May 2018.

This report details observations from the examination of the samples along with the results of analyses carried out, and concludes with comment on the composition of the mortars and the binder type used. In addition, the response to the questions posed by the client relating to the sands and the limestone sample are also included.

2.0 Sample

A batch of samples was received in CMC's Stirling laboratory on the 29th May 2018, with the samples identified as materials from the York Minster Trust. On receipt in the laboratory the sample details were entered into the sample register and the unique sample reference SR2600 allocated. Details of the samples and type are detailed below along with the laboratory and Client sample references:

CMC Sample Reference.	Client Sample Reference.	Location/Comment
SR2600-S1	S1	Nosterfield washed river sand from the river Ure.
SR2600-S2	S2	Sand from the river Ouse, York.
SR2600-S3	S3	15th Century Mortar, from York Minster
SR2600-S4	S4	19th Century Mortar, from York Minster.
SR2600-S5	S5	Highmoor Limestone, proposed for the production of quicklime.

Subsequent to this samples of quicklime, including fully calcined quicklime and an overburnt sample of limestone, prepared from the burning of Highmoor limestone, was supplied by Nigel Copsey, from a trial burn, along with a sample of mortar prepared from this quicklime. On receipt these were logged under the sample reference SR2610 and consisted of the following:

SR2610-S1	A	Two pieces of well, burnt limestone.
SR2610-S2	B	Two pieces of over-burnt limestone.
SR2610-S3	C	Lump of mortar cast using quicklime represented by sample S1.

A sample of the quarried limestone was also provided, however, as this was the same as sample SR2600-S5, i.e. Highmoor Limestone, no analysis was carried out on this sample.

3.0 Methods of Examination and Analysis

The samples were initially photographed on receipt in the laboratory, logged with their mass and size recorded prior to being submitted to an examination with the aid of a stereo-binocular microscope at a magnification up to x 20. During the examination the samples were 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.

A selection of sub-samples was prepared, from each sample, with these submitted to analysis by X-ray Powder Diffraction (XRD). This to aid identification of the binder type employed in each of the mortars, and also assist in clarifying the mineral composition of the sands and limestone samples received.

The mix composition of the mortars was determined by acid digestion, which was carried out following the procedures of the Scottish Lime Centre Trust (SLCT). On completion of the acid digestion the aggregates were recovered by vacuum filtration, dried and graded through a nest of British Standard sieves.

A petrographic thin section was prepared from each mortar sample and from the piece of limestone supplied. This would enable additional information to be obtained, relating to the fabric condition of the mortar, and the form in which the binder was used at the time of mixing. The thin section prepared from the limestone was used to confirm its type and determine if there were any components present that could contribute any hydraulicity to lime produced from this limestone.

Two resin mounted stubs were prepared from the dominant fraction in each sand sample. This was to permit identification of their mineral content and permit proportioning of the dominant components, to assist in clarifying which, if any, had been used in the mortars from each period of works.

The sections prepared were submitted to a petrographic examination in an Olympus BH2 Polarised light microscope, with digital images recorded for record purposes. A selection of the images is included in the report.

4.0 Macroscopic Examination

A summary of the properties of the samples, as received, is presented below:

CMC Sample No.	**Client Ref**	**Mass (gram)**	**Maximum Dimension (mm)**	**Colour – Munsell Soil Colour Chart**	**Moisture Content (%)**
SR2600-S1	Ure Sand	369.1	2.4	2.5Y 6/2 light Brownish Grey	0.3
SR2600-S2	Ouse Sand	336.6	0.55	2.5Y 4/4 Olive Brown	0.9
SR2600-S3	15th C Mortar	155.6	45.6 x 39.2 x 22.1	White	1.2
SR2600-S4	19th C Mortar	600.6	180.1 x 85.5 x 56.0	White	2.2
SR2600-S5	Highmoor L/S	409.4	97.2 x 56.5 x 30.3	10YR 8/2 Very Pale Orange	<0.1

A description of each sample is given in the following section of this report, with details of the samples received from the calcining trial and the mortar made from this, are given in Appendix "A" to this report.

4.1 Macroscopic examination of samples

Sample SR2600-S1 – Nosterfield washed river sand from the river Ure

This sample is fine washed sand, received in a dry condition.

Plates No. 1 & 2: The above left plate shows the sample as received, with the right plate showing a close-up of the sand grains.

The aggregates are dominated by quartz and appear to have a maximum grain size of 3.4mm but mostly finer than 1.0mm and with a low fines content <0.063mm, suggesting a river or dredged source, or a washed sand. The sand contains a mixed suite of minerals dominated by quartz with lithic fragments, including limestone, quartzite and indeterminate igneous rock types. The colour[1] of the sand, was found to be close to 2.5Y 6/2 "Light Brownish Grey".

The result of the grading analysis carried out on the sand sample are given in the table below and as aggregate filled histogram in the appended Figure No. 1.

BS Sieve Size (mm)	**Retained %**	**Passing %**
4.00	0	100
2.00	1.6	98.4
1.00	13.8	84.6
0.500	24.7	59.9
0.250	36.3	23.6
0.125	19.8	3.8
0.063	2.6	1.2
Passing 63µm	1.2	

Table No. 1: Grading of Aggregate from River Ure (SR2600-S1)

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[1] The colour of the mortar and sand samples was determined against the Munsell Soil Colour Charts

Sample SR2600-S2 – Sand from the river Ouse, York

This sample was of another river sand, with this sample having a much finer particle size. The maximum size being 0.34mm with most of the grains finer than 0.2mm. Again, the sand contains a low proportion passing the 0.063mm sieve.

However, unlike sample S1, this sand is dominated by quartz, along with a proportion of limestone and a minor proportion of other lithic fragments.

The colour of the sand was assessed as 2.5Y 4/4 “Olive Grey”, and this sample was also received in a dry condition.

Plates No. 3 & 4: The left plate shows the sample as received, note the very fine grain size compared to sample S1. The right plate shows a close-up of the sand grains, same magnification as in plate No. 2 above.

A grading analysis was carried out on this sand sample with the results obtained reproduced in the table below. The results are also presented in the form of an aggregate filled histogram in Figure No. 2 appended to this report.

BS Sieve Size (mm)	**Retained %**	**Passing %**
4.00	0	100
2.00	0	100
1.00	0	100
0.500	0.1	99.9
0.250	2.2	97.7
0.125	76.7	21.0
0.063	17.4	3.6
Passing 63µm	3.6	

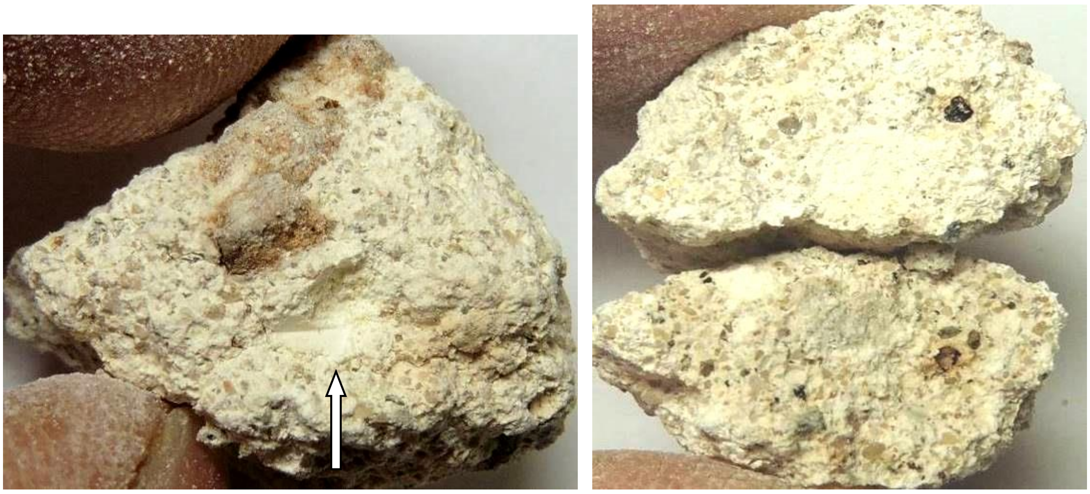
Table No. 2: Grading of Aggregate from River Ouse (SR2600-S2)

Sample SR2600-S3 – 15th Century Mortar, from York Minster

Plates No. 5 & 6: The left plate shows the condition of the sample as received, with the right plate showing an image of one of the larger fragments, in which it can be seen that there is an abundance of small lime inclusions distributed throughout the fabric

The sample pieces appear to be from a well compacted lime mortar containing an abundance of lime inclusions which on measurement were found to range up to 5.8mm in size. The inclusions are angular to sub-angular in shape and can be probed with a point pick suggesting that they are mostly from a well calcined limestone. The mortar is variable in hardness ranging from soft and finger friable to firm and hard requiring persistent firm finger pressure to break and powder.

The mortar was found on comparison with the Munsell Soil Colour Charts to be “White”, with, on testing with a phenolphthalein indicator solution, the mortar found to be fully carbonated.



Plates No. 7 & 8: The left plate shows a magnified view of a freshly fractured surface through one fragment, where an angular lime inclusion can clearly be seen, arrowed in plate. The right plate shows another fracted fragment, after testing the lower piece with a phenolphthalein indicator solution. Where no colour change is observed, this is usually taken to confirm that the mortar is fully carbonated.

Tests with water droplet showed that when the droplets were placed onto surfaces they rapidly absorbed and diffused through the fabric, indicating a well-connected porosity.

Aggregates are sub-angular to sub-round in shape, with sharp margins and are dominated by quartz, with minor limestone and quartzite long with other lithic fragments also present.

Sample SR2600-S4 – 19th Century Mortar, from York Minster

This sample consisted of a single intact piece of mortar, the mortar being well compacted. It was, however, noted that the mortar appeared to be light in weight for its size, and therefore of low density. The pieces could be broken under moderate pressure, and, once disrupted, could be powdered under light to moderate finger pressure.

Plates No. 9 & 10: The left plate shows the intact piece forming the sample, as received. With the right plate showing a close-up of a freshly fractured surface through the mortar fragment. Note the abundance of small lime inclusions distributed throughout the area in view.

Tests with a phenolphthalein solution indicated that the mortar was only surface carbonated. With water droplet tests confirming a well-connected open pore structure, with their rapid absorption, and diffusion throughout the full thickness of the sample tested.

Plates No. 11 & 12: Small sub-round lime inclusions can be seen within the fabric of the mortar in the left plate. The right plate shows a freshly fractured surface on which a phenolphthalein indicator solution had been applied, lower piece. Note the deep fuchsia colour over most of the sample, inferring that the mortar had not carbonated, except at its outer surfaces.

The mortar contains an abundance of irregular to sub-rounded lime inclusions with these extending up to 2.2mm in size. Some of the inclusions are similar in shape and texture to those that appear to have formed from balled hydrate, whilst others have the appearance of entrapped quicklime.

The mortar also displays a patchy air entrainment. There are also areas, adjacent to outer surfaces that are either leached or poorly compacted, with the outer margins also containing an abundance of small entrapped air voids.

The colour of the mortar was found to be “White”.

Aggregates appear to differ from those in sample S3 in that they have a higher limestone and quartz content, along with minor lithic fragments. A proportion of coal fragments and ash clinker was also noted. The sand grain measure up to 5.7mm in size.

Sample SR2600-S5 – Highmoor Limestone, proposed for Quicklime production

The sample of Highmoor limestone consisted of four pieces apparently broken from a piece of sawn masonry, with a number of pieces retaining flat sawn surfaces.

The limestone is compact and hard and required a hammer impact to break and the use of an impact mortar to crush and powder the sample for analysis.

The limestone is 10YR 8/2 “Very Pale Orange”[2] in colour and was noted from a water droplet test to display a degree of microporosity.

Plate No. 13:

The plate on the left shows the condition of the sample of Highmoor Limestone as received.

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[2] Geological Society of America – Rock Colour Chart

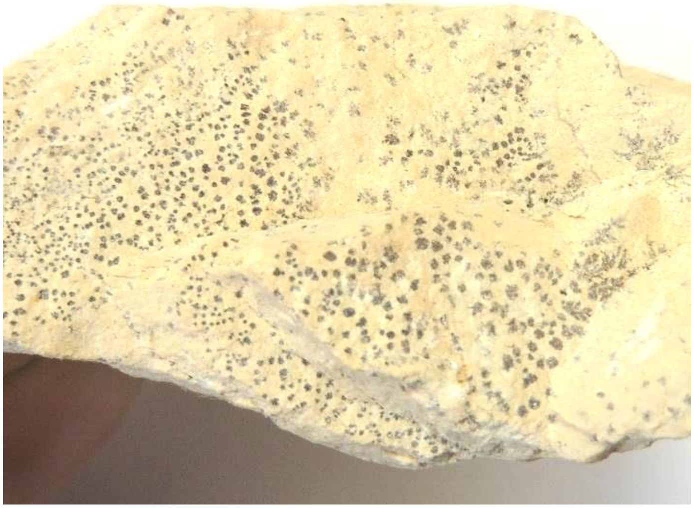

Plate No. 14:

Close-up of a freshly fractured surface, showing a mottling of organic material within the rock fabric, this is not uncommon in this form of limestone.

5.0 Microscopic Examination

Petrographic thin sections were prepared from slices sawn from the two mortar samples, with the selected slices aligned to permit the maximum area of the mortar, on the slides.

The mortar samples were prepared for thin sectioning by initially drying them to a constant weight at 60°C prior to impregnating the dried sub-samples with an epoxy resin containing a fluorescent blue dye.

One side the impregnated slices were polished and mounted onto glass slides (50mm x 75mm). The mounted samples were then ground and polished to a thickness of approximately 30microns.

A representative, quartered sub-sample, from the dominant fraction in each of the sands was dried and cast into a mould and bound in a blue dyed polyester resin. Once set a slice was cut through the diameter of the castings and these mounted on glass slides, as for the mortar samples.

A slice cut through the limestone sample was also prepared for microscopic examination, with half the area tinted to aid identification of dolomite in the stone.

The microscopic examination of the thin sections was undertaken using an Olympus BH2 Polarised light microscope, fitted with a Digital Camera, to permit the recording of images of areas of note for record purposes.

The presence of dyed epoxy resin enables detailed analysis of void distribution, an assessment of microporosity and a clear indication of any crack patterns present, in plane polarised light.

The samples were also mounted onto a ‘Swift automatic stage’ which permitted the determination of the volumetric mix composition, by modal analysis.

The observations from the examination of the samples are presented below:

5.1 Sample SR2600-S1 – Nosterfield washed river sand from the river Ure

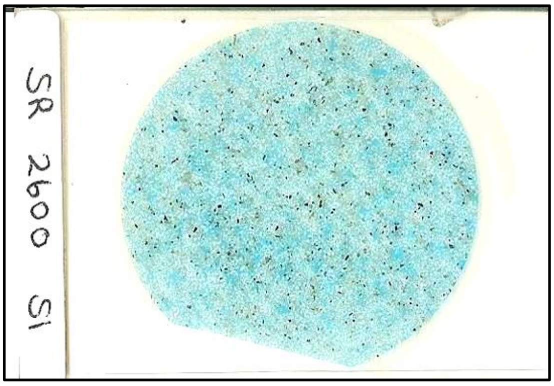

Plate No. 15:

The left plate shows the thin section prepared from the dominant fraction (passing 0.500mm, retained 0.250mm) in sample S1, sand from the River Ure.

The sand grains are between 0.25mm and 0.625mm (medium to coarse sand) and comprise significant proportions of quartz (48.8%) and limestone (33.3%).

The limestone component includes fossil fragments and calcite cement, which are derived from limestone and some of the limestone is dolomitic (approximately 1.5% of the total constituents).

Other components include chert (5.8%), which is likely to be derived from the silicified parts of the source limestone. Sandstone and quartzite (6.2% and 3.3% respectively) are quartz dominated, where grains are either tightly fused by silica cement and point contact dissolution (quartzite) or cemented together by a combination of cementing agents that include clay minerals, silica and iron oxides/ hydroxides (sandstone).

Minor feldspar (1.1%) is composed of alkali and plagioclase, which appears fresh and unaltered. Minor opaque minerals (1.1%) are considered to be iron bearing particles, derived from both the sandstone and limestone source horizons.

Grains are sub-rounded to sub-angular and have the appearance typical of a natural sand, with the rounding indicative of water transport, suggesting a river source.

Components	S1
Quartz	48.8
Limestone	31.8
Sandstone	6.2
Chert	5.8
Quartzite	3.3
Dolomite	1.5
Feldspar	1.5
Opaque Particles	1.1
Clay/Shale	0.0
Total	100.0

Table No. 3: Proportion of the minerals present in the dominant fraction (<500 µm >250µm)

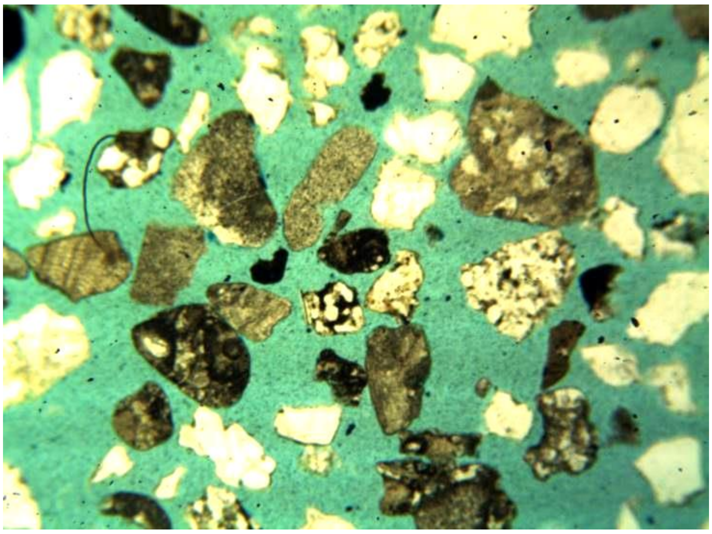

Plate No. 16:

A view in plane polarised light (ppl), of a typical area of the slide.

Most of the aggregates in view are quartz (white in the plate) along with limestone, quartzite, sandstone and chert, with minor opaque and feldspar.

Field of view 2.4mm.

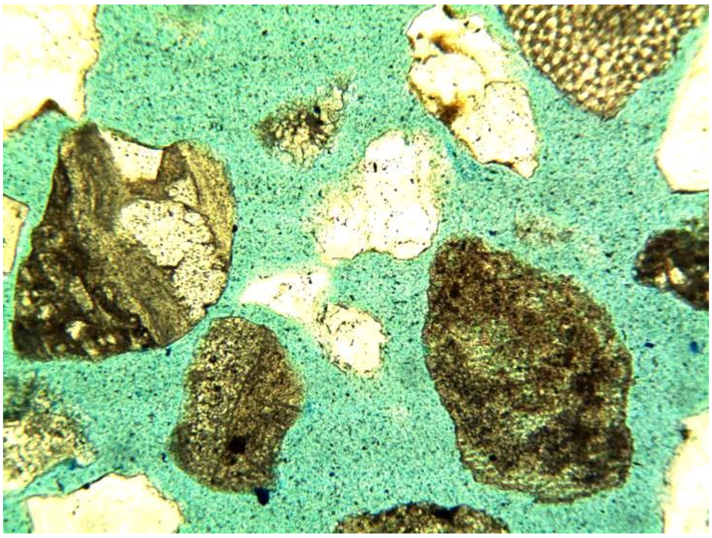

Plate No. 17:

Another view in plane polarised light (ppl), of an area of slide.

The aggregates in view include limestone (left and right lower) quartz (centre), sandstone (left) and Dolomite (upper right)

Field of view 1.2mm.

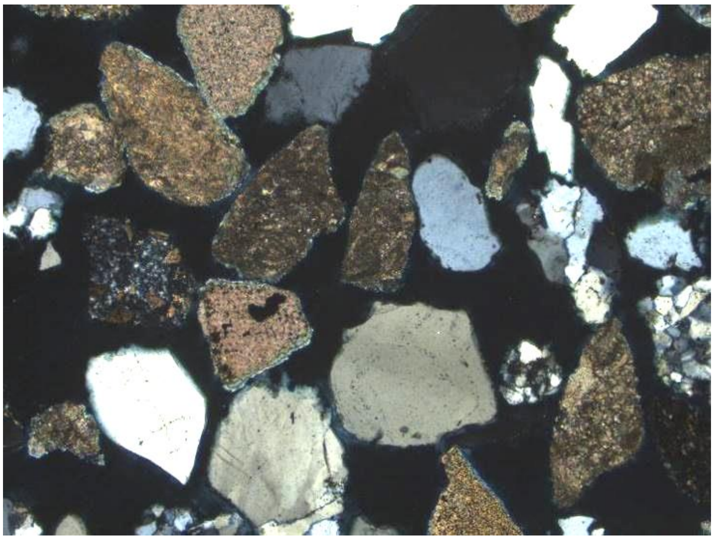

Plate No. 18:

A view in cross polarised light (xpl), of a typical area of slide.

The aggregates in view are quartz, limestone, quartzite, sandstone and chert, with minor opaque and feldspar

Field of view 2.4mm.

The impregnating resin and opaque minerals all appear black in xpl.

5.2 Sample SR2600-S2 – Sand from the river Ouse, York

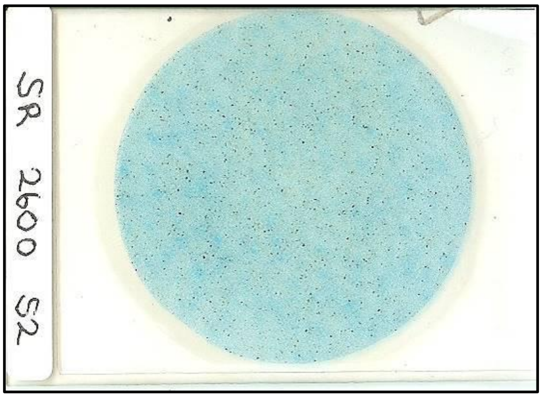

Plate No. 19:

Thin section prepared from the dominant fraction (passing 0.250mm, retained 0.125mm) in sample S2, sand from the River Ouse, in York.

The grains in this sand sample are between 0.1mm and 0.3mm in diameter (very fine to medium grained sand) and range in shape from sub-rounded to angular. They are generally more angular and smaller than the grains observed in S1.

The sand is dominated by quartz (85.4%) and this comprises monocrystalline unstrained to locally strained varieties. Polycrystalline quartz is minor. Quartz overgrowths are commonly preserved and many of these quartz grains are derived from sandstone, where other cementing materials, including clay minerals and iron oxides, are retained on the grain surfaces. The other components are present in minor amounts and dominated by siliceous types. Sandstone (3.6%) is quartz rich and comprises quartz grains cemented by silica and clay minerals. Chert (3.0%) is composed of microcrystalline silica and is of sedimentary origin.

Minor quartzite is composed of fused quartz crystals, most likely, given the locally preserved clay coatings, to be derived from a sandstone source. Alkali and plagioclase feldspar (3.6%) include both alkali and plagioclase varieties that are slightly altered to sericite along cleavage planes. Minor clay and shale particles are laminated fragments, and some appear soft and weak. However, some of these contain opaque minerals and possibly microcrystalline silica. The opaque particles (0.7%) in this sample locally contain quartz sand grains and are considered to be of a sedimentary origin.

Components	S2
Quartz	85.4
Limestone	0
Sandstone	3.6
Chert	3.0
Quartzite	0.7
Dolomite	0
Feldspar	3.6
Opaque Particles	0.7
Clay/Shale	3.0
Total	100.0

Table No. 4: Proportion of the minerals present in the dominant fraction (<250 µm >125µm)

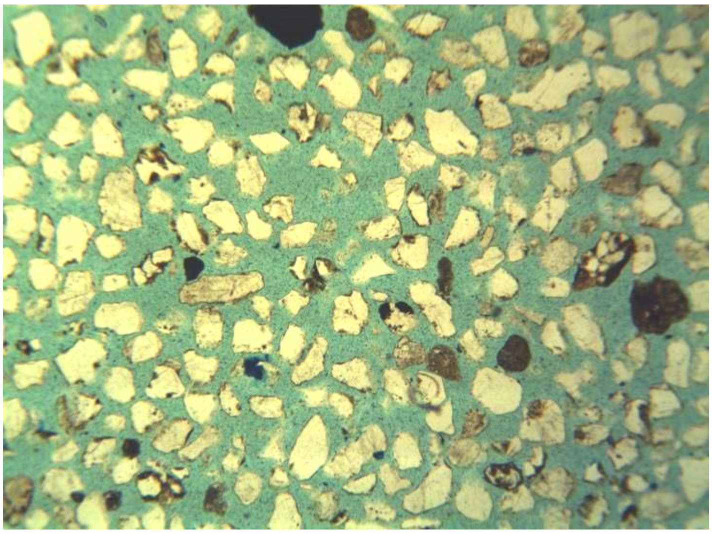

Plate No. 20:

A view in plane polarised light (ppl), of a typical area of slide.

This image is at the same magnification as that in Sample S1, for direct comparison of grain size and shape.

Most of the aggregates in view are quartz (white in the plate), sandstone fragments with minor chert, opaque and feldspar minerals

Field of view 2.4mm.

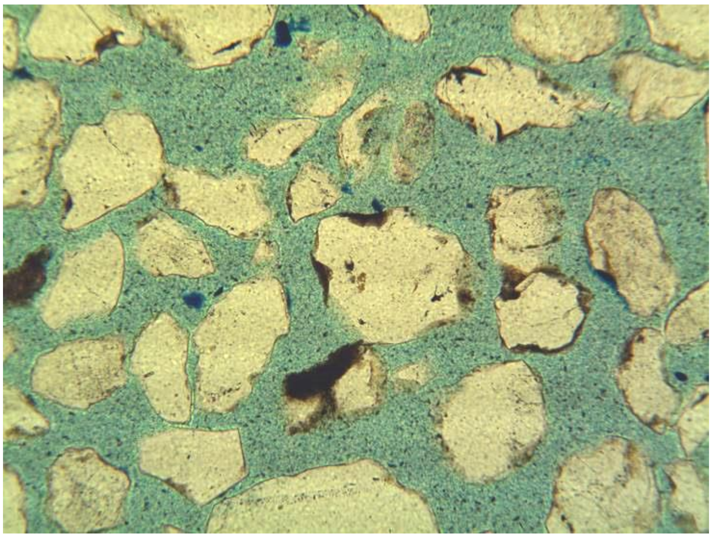

Plate No. 21:

Another view in plane polarised light (ppl), of an area of slide.

The aggregates in view are mostly quartz (centre), some of which retain opaque coatings

Field of view 1.2mm.

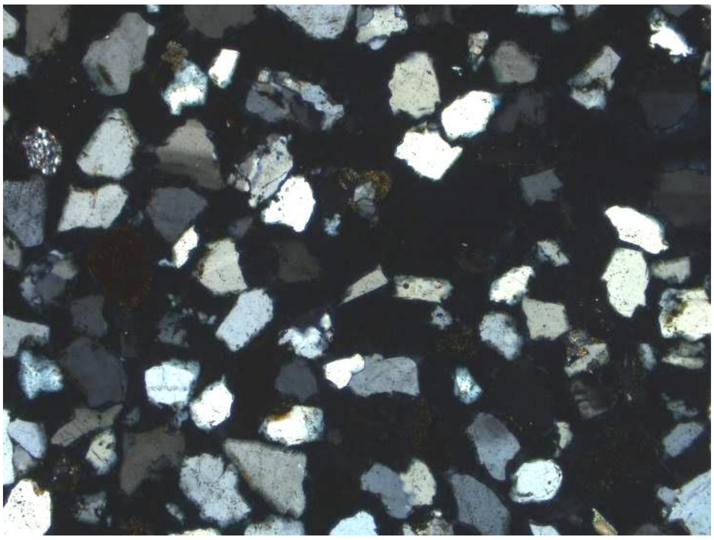

Plate No. 22:

A view in cross polarised light (xpl), of a typical area of slide.

The aggregates in view are quartz, quartzite, sandstone and chert, with minor opaque and feldspar minerals.

Field of view 2.4mm.

The impregnating resin and opaque minerals all appear black in xpl.

5.3 Sample SR2600-S3 – 15th Century Mortar, from York Minster

The thin section was prepared from the largest intact piece of mortar in the sample.

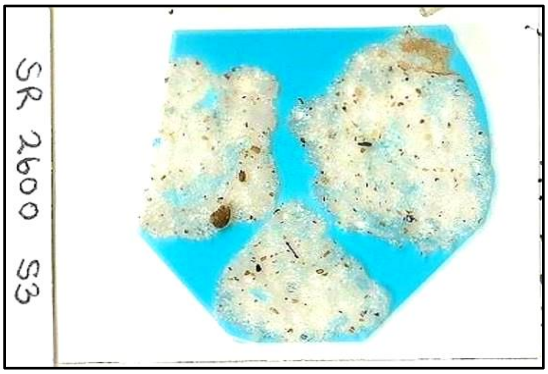

Plate No. 23:

This thin section was prepared from three pieces of mortar from sample S3 for microscopic examination.

Aggregate

The aggregate is composed of a mixed suite of minerals, dominated by quartz and limestone along with feldspar and altered lithic (rock) fragments. The grains are sub-angular to sub- round, and irregular in shape, although the grains display partial rounding at their margins, indicative of water transport. The grains are between 0.24mm and 1.4mm in size and quartz accounts for 59.6% of the aggregate grains present, with limestone (22.8%) and minor sandstone (5.3%), quartzite (3.7%), chert (3.1%), Dolomite (1.4%), feldspar (1.2%) and opaque minerals (2.9%).

The aggregates are well bound within the paste and although some peripheral cracking is apparent, these are mostly discontinuous and have the appearance of early plastic and drying shrinkage features and are considered unlikely to have an impact on the performance of the mortar.

A low proportion of the limestone fragments apparent in aggregate show dolomitic textures along with burnt margins, and this may infer that they had been included in the mortar, along with the quicklime, as unburnt feedstock from the kiln, rather than with the aggregate.

Binder

The binder is of a non-hydraulic lime, with the mortar noted to contain an abundance of incompletely burnt and incompletely mixed, but fully slaked, quicklime inclusions, along with a low proportion of overburnt particles also present.

The lime inclusions are sub-angular to sub-round in shape and their appearance and texture would suggest that the quicklime had been slaked with the sand, and not prepared as a hydrate or run to a putty before use. The compactness of the paste around some inclusion margins, along with adjacent clusters of small spherical voids within the encapsulating paste, and the low abundance of shrinkage cracks and open channel ways in the mortar fabric, inferring that the mortar had probably been mixed, and placed, as a Hot Mixed Mortar (HMM).

The paste in the mortar is fully carbonated, and locally there is some evidence of loss of paste by leaching, which along with the presence redeposited lime within voids and some wider cracks, would suggest water percolation thought the mortar in the past.

There is also localised alteration of the fabric to suggest that some of the lime inclusions had slaked late, with disruption, and loss, of the core material, perhaps in response to water percolation through the fabric, after placing, along with age related weathering. A number of the incompletely calcined lime inclusions contain cores retaining an imprint the original rock fabric, with these confirming that, at least, a proportion of the limestone burnt in lime production was Dolomitic, with re-formed dolomitic rhombs observed within fully carbonated intact slaked lime inclusions.

There is no evidence of alteration products within the body of the mortar, and no evidence of disruptive chemical attack, i.e. sulphate, etc. and the fabric of the mortar appears sound.

Voids and microcracks

Voids are abundant and are present, both as angular to irregular shaped voids formed as placing artefacts, and from the dissolution of inclusions, ranging up to 2.7mm in size. A proportion of small discrete entrained and entrapped air bubbles (typically <0.2mm in size) were also observed within the paste, most notably in dense patches of matrix.

Cracks are rare, and where present are of two forms, as branching cracks emanating from inclusions and as random shrinkage cracks. They range in width from <0.02mm to 0.04mm. The cracks and voids are mostly free of secondary minerals, albeit there is localised evidence of fluid migration through the mortar, with fine fringes of redeposited calcite observed.

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

Sample Ref:	**SR2600-S3**	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as aggregate**
Quartz	22.7	22.7
Limestone	8.4	8.4
Lithic rock fragments	5.9	5.9
Opaque Minerals	1.1	1.1
Lime Inclusions & Clinker	-	20.1
Total Aggregate	**38.1**	**58.2**
Binder (Lime Paste)	39.9	39.9
Clinker	0	0
Lime Inclusions	20.1	-
Secondary products/Calcite	1.9	1.9
Total Binder	**61.9**	**41.8**
Total Constituents	**100.0**	**100.0**
Voids	6.3	6.3
Crack	1.3	1.3
Cracks/Voids	**7.6**	**7.6**
Mix Composition, by volume		
Binder: Aggregate Ratio	**Total Binder**	**Effective Binder**
	1.0 : 0.62	**1.0 : 1.4**

Table No. 5: Modal Analysis carried out on thin section prepared from sample S3.

The effective binder content is calculated on the basis that the inclusions are acting as aggregate rather than as 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 at the time the mortar was made and placed, including the inclusions (both fully slaked and unslaked) as part of the added lime binder.

Photomicrographs:

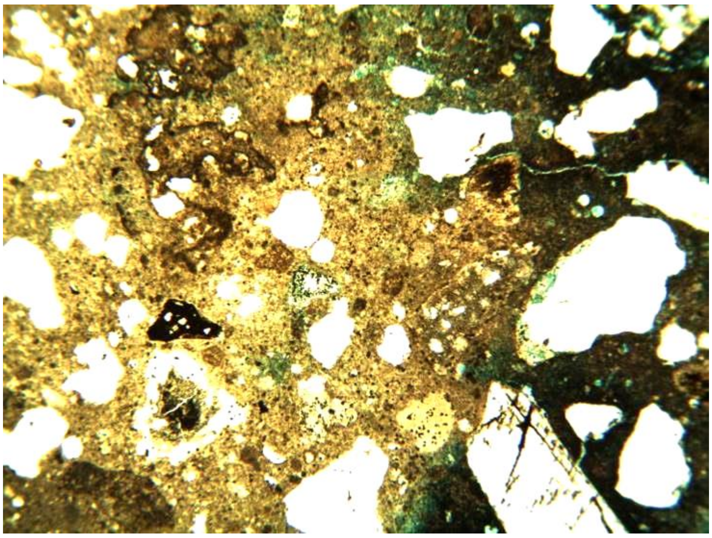

Plate No. 24:

A view in plane polarised light (ppl), of a typical area of the mortar where the paste on the left side of the plate is more microporous, compared to the dense paste to the right, all of the paste is fully carbonated.
Most of the sand grains in view are quartz (white in section), with various lithic fragments also apparent. Limestone aggregate can be seen in the lower left and centre right, the latter being dolomitic.
Porosity is highlighted by the blue dyed resin.
Field of view 2.4mm.

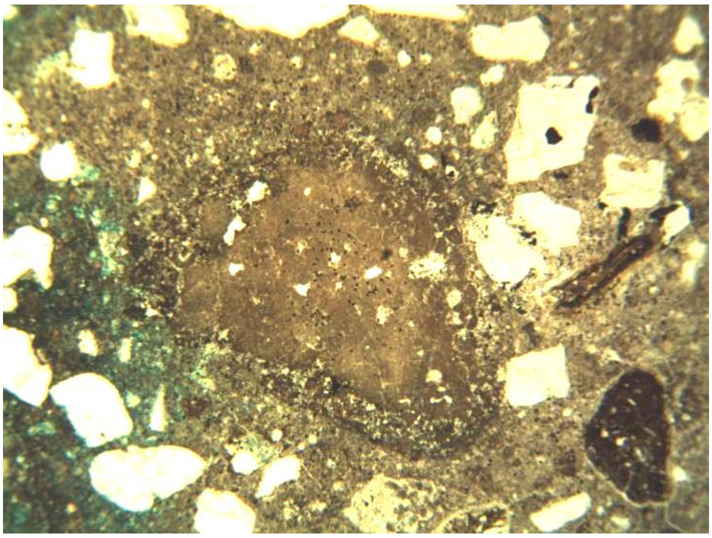

Plate No. 25:
Another view in ppl, of an area of paste containing a lime inclusion, centre of plate. The inclusion is only partially calcined with only the outer margin having slaked. Note the dense margin around the inclusion, which has only partially diffused into the surrounding paste. This inclusion has the appearance of slaking in place, after encapsulation within the mortar fabric, as in a hot mixed mortar (HMM).
Aggregates are again dominated by quartz.
Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

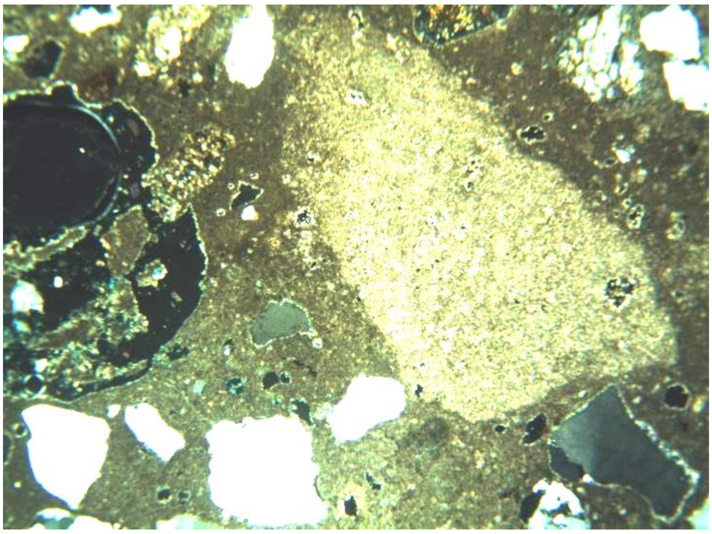

Plate No. 26:
A view in cross polarised light (xpl) of an area of paste containing a lightly burnt limestone fragment, right side of plate and a partially filled void containing remnants of a slaked and depleted lime inclusion, left side of plate. The limestone is dolomitic and locally displays evidence of alteration, the upper left edge has partially merged into the paste. The void on the left retains fragments of an overburnt inclusion, with the void formed rimmed with redeposited calcite. Aggregate grains are again dominated by quartz.
Voids and the blue dyed resin appear dark in cross polarised light. Field of view 2.4mm.

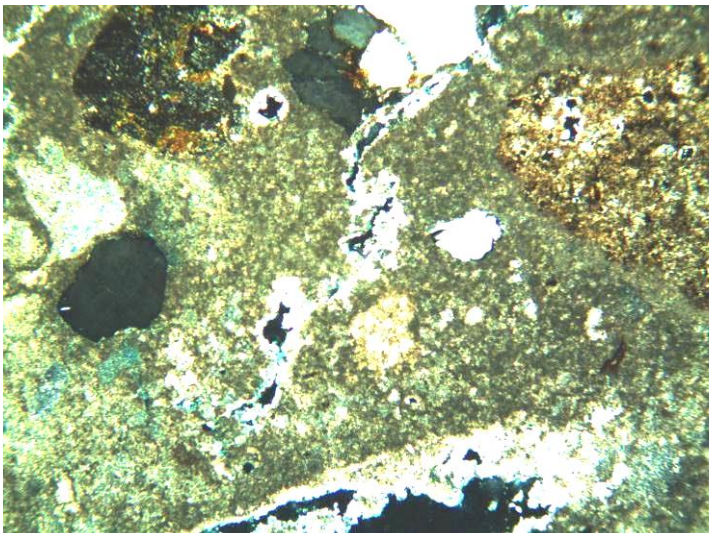

Plate No. 27:
Another view in cross polarised light (xpl) of dense area of paste through which a crack extends, this has acted as a fluid percolation pathway and is rimmed with redeposited calcite.
At the base of the plate a void can be seen which retained a thick crust of calcite and contains the remnants of a disrupted lime inclusion.
Further sub-rounded voids, free of coatings can be seen in the left and upper left in the plate, these may be air voids, or from steam, generated from the lime slaking.
Voids and the blue dyed resin appear dark in cross polarised light. Field of view 1.2mm.

5.4 Sample SR2600-S4 – 19th Century Mortar, from York Minster

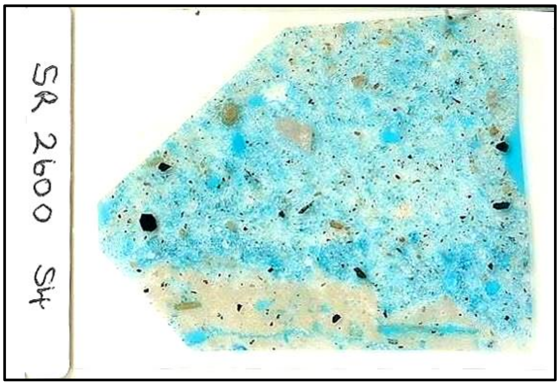

Plate No. 28:

The thin section opposite was prepared from an intact piece of the mortar from sample S4 for microscopic examination and determination of mix composition, by modal analysis.

This sample consisted of a large piece of mortar bonded to a thin slice of limestone masonry, which can be seen in the lower part of the slide above.

Aggregate

The aggregates in the mortar sample consist of a mixed suite of rock types, which is dominated by quartz (64.5%), along with quartzite (2.3%), sandstone (2.7%), limestone 24.6), chert (1.8%) and minor feldspar (1.2%) and opaque minerals (2.9%).

The presence of a proportion of coal ash and clinker, (15.1%) along with some wood ash was also observed, and its inclusion at a significant proportion would infer that the former had probably been added as a pozzolan, rather than as a contaminant of the aggregate, or the quicklime. The wood ash may have been added with the lime or be a contaminant in the aggregate. Although, it has been reported that proportions of wood ash were added to some lime mortars to improve wet weather performance and aid curing.

The aggregates are sub-angular to sub-round in shape, with a low proportion of elongated aggregate particles also present. The shape of the grains, along with the sharp margins and texture of the aggregates suggesting a pit deposit as the source for the sand.

The aggregates are mostly well bound within the paste and the aggregate grains range in size from 0.04mm to 1.7mm (coarse silt to coarse sand).

Binder

The binder is typical of a lime mortar, with the presence of lime inclusions apparent within the section. The inclusions appear to have formed from non-hydraulic quicklime, which appear to have been well burnt and fully slaked. Most of the intact inclusions observed are fully slaked and have the appearance of uncarbonated hydrate, however, there is a low abundance of under burnt inclusions available, which retain a faint imprint of the original rock fabric, with these confirming that a proportion of the limestone was Dolomitic or Magnesian limestone, some of the fragments appear calcareous and are from an oolitic limestone, partially dolomitised.

The paste is only partially carbonated, with this appearing as invasive carbonation extending in from the perimeter of the sample and adjacent to the limestone fragment, inferring that the sample lump may have separated, from the wall mass along pre-existing cracks, that had acted as fluid migration channel-ways, prior to sampling.

The lime inclusions apparent, include both sub-round and sub-angular inclusions, with most having the appearance of having fully hydrated, with these having an internal texture in common with that observed in the paste. It is, therefore, indicated that the binder was probably hydrated prior to use, but not fully screened. With the presence of an abundance of angular fragments, along with a proportion of partially burnt and partially hydrated lime, it is probable that the lime was slaked with the sand, and slaked to a dry condition, prior to being remixed in preparation for placing, with it probably used and placed as a cold mixed mortar.

The abundance of ash in the mix, some of which appears to have altered, with coarser particles displaying reaction rims, thereby inferring that a pozzolanic reaction had occurred, imparting a measure of hydraulicity to the mortar. The pozzolan being added, in the sample examined, at a dosage in the region of 1 part binder to 0.2 parts ash.

Voids and microcracks

Voids are present, and these are typically round to sub-round in shape and range from 0.08mm to 1.4mm in size, and appear to be water formed voids, with most retaining a fine fringe of Portlandite and calcite.

Cracks are rare and occur in clusters, mostly as localised features peripheral to the outer zones of the piece sampled. The cracks range in width from <0.01mm to 0.03mm and are typical of early drying shrinkage features.

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

Sample Ref:	**SR2600-S4**	
Constituents	%	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	34.2	34.2
Lithic grains	4.4	4.4
Limestone	13.5	13.5
Opaque Minerals	2.9	2.9
Ash/clinker	8.3	8.3
Lime Inclusions & Clinker	-	3.4
Total Aggregate	63.3	66.7
Binder (Lime)	33.2	33.2
Hydraulic Clinker	0	0
Lime Inclusions	3.4	-
Secondary products/Calcite	0.1	0.1
Total Binder	**36.7**	**33.3**
Total Constituents	**100.0**	**100.0**
Voids	8.3	8.3
Crack	0.7	0.7
Cracks/Voids	**9.0**	**9.0**
Binder: Aggregate Ratio	**Total**	**Effective**
	1.0 : 1.7	**1.0 : 2.0**

Table No. 6: Modal Analysis carried out on thin section prepared from sample S4.

Photomicrographs:

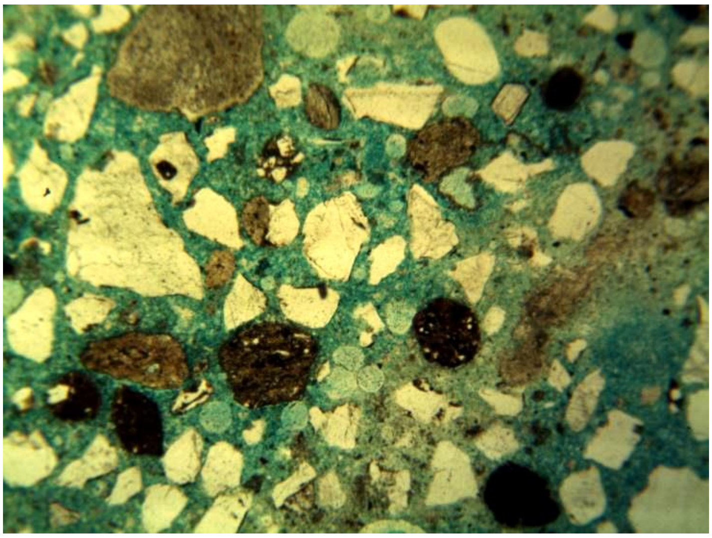

Plate No. 29:

A view in plane polarised light (ppl) showing a typical area of the mortar containing a mixed suite of minerals in the aggregate, and although dominated by quartz; limestone, sandstone and quartzite are also present. Ash clusters can also be seen randomly distributed throughout the paste.

Most of the paste in view is uncarbonated with a partially carbonated band transecting the right side of the plate, along with an area of depleted paste in the lower right.

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

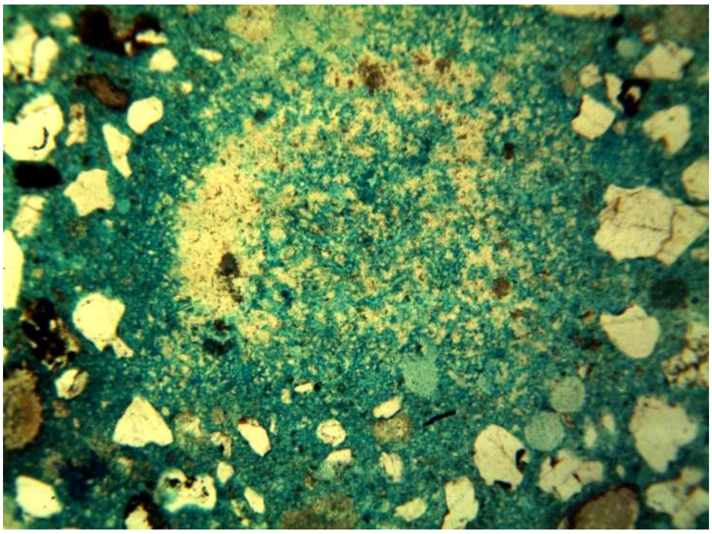

Plate No. 30:

A partially depleted lime inclusion, in ppl, where the outer margin of the inclusion can be seen along with relic fabric texture in the centre left.

The limestone burnt was dolomitic and there is evidence of rhombs within the unslaked fabric along with darker areas which are considered to be associated with organic matter in the limestone.

The paste, surrounding the inclusion is uncarbonated.

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

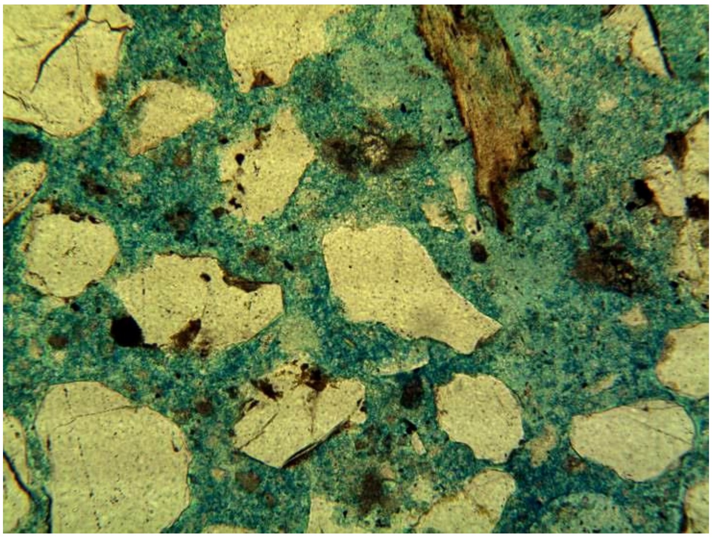

Plate No. 31:

Another view in plane polarised light (ppl), of an area containing an abundance of ash distributed throughout the area, seen as grey, brown and black in the image. Diffused margins around some of these particles inferring that a reaction has occurred.

Aggregates are dominated by quartz, with feldspar, with the former retaining patches of opaque coatings. A fragment partially burnt wood can be seen upper right.

Porosity is highlighted by the blue dyed resin. Field of view 1.2mm.

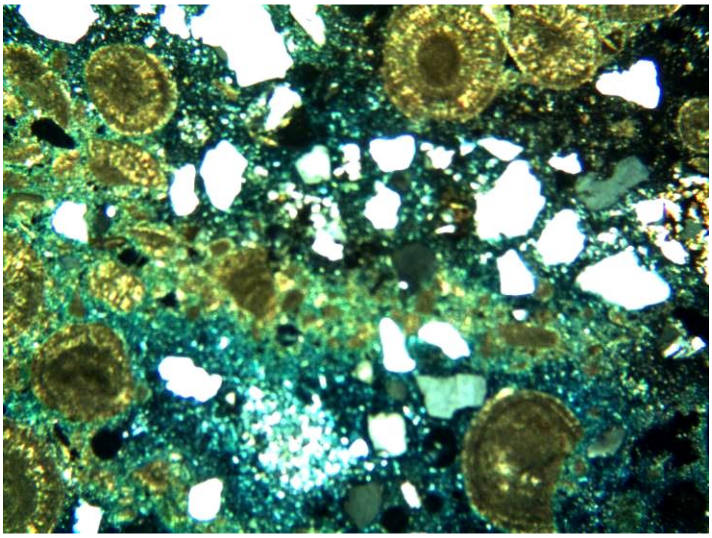

Plate No. 32:

A view in cross polarised light (xpl), of an area containing a cluster of ooliths, which appear to have originated from a coarse limestone fragment that has broken down within the mortar. Aggregates are dominated by quartz, white in plate.

The paste in the upper part of the slide and in the lower centre and right is uncarbonated, whereas that in contact with the limestone dust and ooliths, centre and lower left, shows the effect of carbonation

Voids and the blue dyed resin appear dark in cross polarised light. Field of view 1.2mm.

5.5 Sample SR2600-S5 – Highmoor Limestone, proposed for Quicklime production

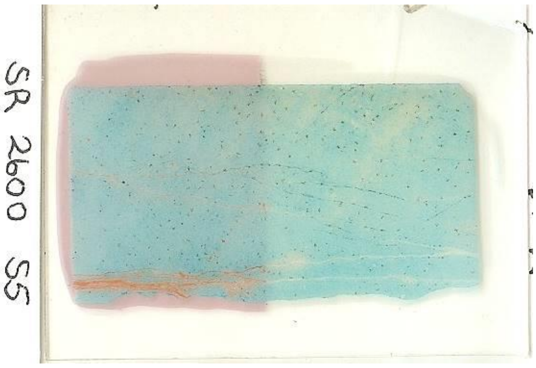

Plate No. 33:

The adjacent plate shows the thin section prepared from a piece of limestone, cut from Sample S4 for microscopic examination.

The rock is composed of dolomitised limestone (dolomite[3]), where dolomite crystals form >95% of the constituents. The texture of the dolomite ranges from a fitted fabric (xenotopic), where the rhombic margins of crystals are sutured, to clearly defined rhombs (idiotopic texture).

The original limestone was oolitic, where calcareous spheres formed the majority of the rock. The sphere rims, during geological processes, have been selectively replaced by dolomite and the original calcite/aragonite of the ooliths dissolved, leaving common voids. The original ooliths were between 0.08 and 0.012mm in diameter and the rims of these are replaced by fine dolomite crystals in the form of fine lacy mats.

Some of the voids are filled with non-ferroan calcite cement, but this material is minor and isolated.

Narrow (0.04mm - 0.12mm) veins of non-ferroan, to locally ferroan calcite, transect the rock and these features are likely to be roughly parallel to the original bedding surface. Indeed, discontinuous concentrations of organic layers of a similar orientation are also likely to be aligned to the bedding orientation. Both of these features represent places of weakness along which fracturing has preferentially occurred.

Minor and scattered concentrations of organic material, locally reaching 0.3mm in diameter, surround cubic opaque minerals (0.06mm), which are likely to include pyrite.

Rare angular quartz sand grains are scattered and range from between 0.1mm and 0.2mm (very fine to fine grained sand).

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[3] Limestone – 0-10% dolomite
Dolomitic limestone – 10 to 50% dolomite
Calcitic dolomite - 50 to 90% dolomite
Dolomite (dolostone) – 90 to 100% dolomite

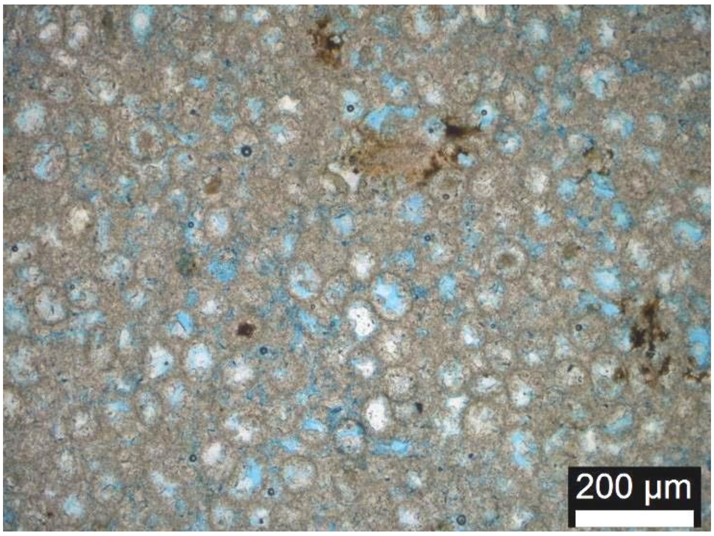


Plate No. 34:

A view in plane polarised light (ppl), of a typical area on the slide.

The area in view shows the remnants of the original oolitic fabric, with the scattered dark concentrations of organic matter seen within the upper and right parts of the plate.

A cubic pyrite crystal can be seen lower left of centre.

Porosity is highlighted by the blue dyed resin.
Magnification is indicated by the scale bar.

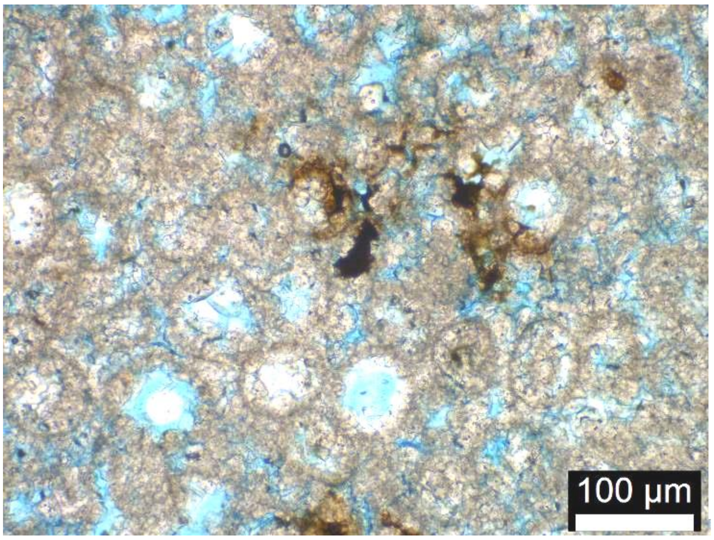


Plate No. 35:

A magnified view in plane polarised light (ppl), of an area of the fabric containing entrapped organic material, black to dark brown in the plate.

Small cubic pyrite crystals can be seen upper right, and right of centre

Porosity is highlighted by the blue dyed resin.

Magnification is indicated by the scale bar.

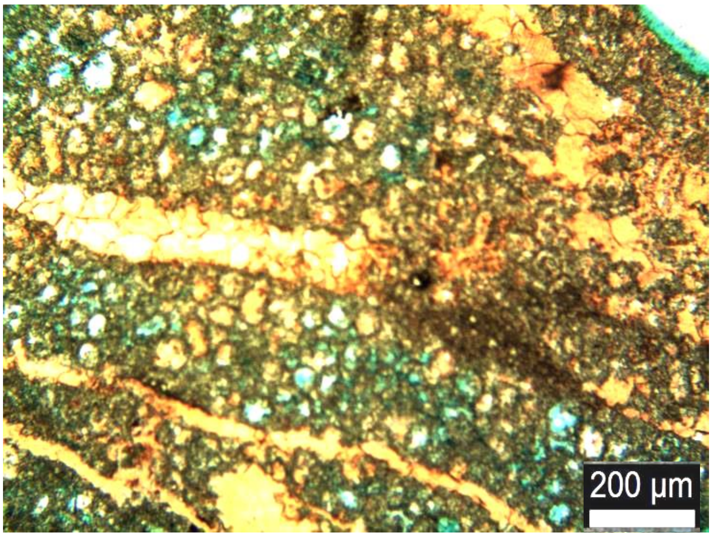


Plate No. 36:

A magnified view in cross polarised light (xpl), of an area of the slide stained to highlight presence of dolomite.

Veins of non-ferroan, to locally ferroan calcite, transect the rock, and these are likely to occur parallel to the original bedding.

Magnification is indicated by the scale bar.

6.0 Analysis by X-Ray Diffraction

To assist in the identification of the composition of the binder in the mortar samples, a binder rich sub-sample from each sample was prepared and these submitted to analysis by X-ray Powder Diffraction (XRD).

To achieve this, a representative sub-sample of the mortar was obtained, from each sample, with these ground in an agate mortar and pestle, taking care to minimise crushing of the aggregate. A concentrated binder rich sub-sample was obtained by sieving the sample over a 63µm sieve. The powder collected was then back-packed into proprietary sample holders for presentation in the X-Ray Diffractometer. This technique being used to ensure the random orientation required to give true peak intensities in the diffractograms.

In addition, a sample of the limestone was also prepared and analysed by XRD to confirm the minerals present and to confirm if it was dolomitic. With a further sample of the limestone calcined in a laboratory Muffle Furnace, with samples raised to both 750°C and 950°C, and the products analysed after ignition and after slaking, to assess if the product produced, at both temperatures. This would also confirm if limestone contained any minerals that could contribute a hydraulicity to the binder.

The prepared samples were 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. With the digital output from the diffractometer analysed in 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 obtained are shown in the following figures appended to the end of this report, in the form of a labelled X-ray diffractograms:

Figure No. 5 – SR2600-S3 – Binder from 15th Century Mortar ex York Minster,
Figure No. 6 – SR2600-S4 – Binder from 19th Century Mortar ex York Minster,
Figure No. 7 – SR2600-S5a – Highmoor Limestone, as received,
Figure No. 8 – SR2600-S5e – Highmoor Limestone, after Calcining at 750°C,
Figure No. 9 – SR2600-S5f – Highmoor Limestone after Calcining at 950°C,
Figure No. 10 – SR2600-S5g – Highmoor L/stone, calcined at 750°C, after Slaking,
Figure No. 11 – SR2600-S5h – Highmoor L/stone, calcined at 950°C, after Slaking,
Figure No. 12 – SR2600-S5j – Highmoor L/stone, calcined at 950°C, slaked and carbonated.

The most abundant mineral components identified are indicated in the appended diffractograms using the following short-hand notation:

li = Lime (CaO) Dominant component from calcining Calcareous limestone,
pe = Periclase (MgO) Magnesium Oxide, Dominant component from the calcining of Dolomitic Limestone,
po = Portlandite ($Ca(OH)_2$) Calcium Hydroxide, hydrated lime and from uncarbonated slaked lime binder,
br = Brucite ($Mg(OH)_2$) Magnesium Hydroxide, hydration product in a dolomitic lime,
cc = Calcite ($CaCO_3$) Calcium Carbonate, carbonated lime binder component, also the dominant component of any limestone in the aggregate,

do = Dolomite ($Ca,Mg(CO_3)_2$) Calcium Magnesium Carbonate, dominant component of Dolomitic limestone and in carbonated Dolomitic lime binders,
ak = Akermanite ($Ca_2MgSi_2O_7$) Calcium Magnesian Silicate, component from calcining dolomitic/magnesian limestone containing silicates, hydraulic component,
mo = Monocarbonate ($Ca_4Al_2O_6CO_311H_2O$) Calcium Aluminium Oxide Carbonate Hydrate, Carbonated *afm* phase, hydration product in a hydraulic lime,
he = Hemicarbonate ($Ca_4Al_2O_6(CO_3)_{0.5}(OH)n11.5H_2O$) Calcium Aluminium Oxide Carbonate Hydroxide Hydrate, *afm* phase, hydration product of a hydraulic clinker,
zo = Zoisite ($Ca_2Al_3(SiO_4)(SiO_7)(O.OH)_2$) Calcium Aluminium Silicate Hydroxide (epidote), clinker component, from burning dirty Limestone, also found in some coal ash/clinker,
ep = Epidote, Calcium Aluminium Iron Silicate Hydroxide, low temperature metamorphic mineral, can be found in some limestones and sandstones,
ma = Magnesite ($MgCO_3$) Magnesium Carbonate, carbonated Periclase & Brucite, present within veins in the Limestone, also rapid carbonation product after hydration,
ett = Ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}26H_2O$) Calcium Aluminium Sulphate Hydroxide Hydrate, hydration product in an aluminous hydraulic/pozzolanic lime,
qz = Quartz (SiO_2) dominant component of the sand grains in the aggregates used in all of the mortar samples, and dominant mineral present in the sandstone sample.

From the analysis it is indicated that the binders used in each of the mortars differ significantly with that used in sample S3 (15th *C* mortar) made using a dolomitic limestone that was relatively pure, producing a non-hydraulic lime. Whereas, although the binder in S4 (19th Century mortar) was also made from a dolomitic or Magnesian lime, the mix was also found to contain a proportion of a pozzolan, in the form of an ash.

The 19th C mortar is not fully carbonated, and the carbonation is limited, with both Portlandite and Brucite present in significant proportions. This may infer that the mortar was either relatively young, or it had been maintained in a damp condition, or there was restricted air flow through the construction in which it was encapsulated, since the mortar was placed. Either of the latter two conditions could inhibit the development of carbonation.

The limestone submitted for analysis is an almost pure dolomitic lime, but with minor veining, that may contain components that could introduce a feebly hydraulicity, i.e. aluminates and/or silicates. The limestone calcined to Periclase and Lime, which on slaking formed Brucite and Portlandite. After carbonation these should revert to Dolomite and Calcite. It was, however, noted that on slaking the quicklime, the Lime readily slaked to Portlandite, but the Periclase took much longer to convert to Brucite. Also carbonation of the Magnesium Carbonate appeared to take much longer. The presence of magnesite may be associated with the veining in the limestone, with the Akermanite detected in the 15th Century mortar, also likely to be a contaminant in the limestone.

The data from the XRD analysis was further processed by Rietveld Refinement. This permitted quantification of the components present and was used to determine hydraulicity of the mortars, and permit comparison between the crystalline components present before after calcining the limestone, and in the hydration and carbonation products formed.

The results obtained are shown below:

Proportions by Rietveld Refinement.

Material:	**15th C**	**19th C**	**Highmoor Limestone**					
	Mortar	**Mortar**	**limestone**	**Quicklime**		**Hydrate**		**Carbonate**
				750°C	**950°C**	**750°C**	**950°C**	**7 day**
Sample Ref:	**S3**	**S4**	**S5a**	**S5e**	**S5f**	**S5g**	**S5h**	**S5J**
			% by Mass					
Lime	-	-	-	10.2	56.0	-	-	-
Periclase	-	-	-	29.1	42.1	42.3	32.1	25.5
Portlandite	-	16.6	-	-	-	57.7	59.9	15.6
Brucite	-	30.6	-	-	-	-	-	3.2
Calcite	49.0	32.8	2.4	60.8	-	-	1.8	52.1
Dolomite	38.8	0.5	97.6	-	-	-	-	-
Magnesite	-	-	-	-	-	-	6.2	3.6
Quartz	7.6	11.8	-	-	-	-	-	-
Akermanite	0.5	-	-	-	-	-	-	-
Mono-carbonate	2.7	-	-	-	-	-	-	-
Hemi-carbonate	1.4	4.5	-	-	-	-	-	-
Zoisite/Epidote	-	0.5	-	-	2.0	-	-	-
Ettringite	-	2.7	-	-	-	-	-	-

Samples set aside to carbonate have still not fully carbonated and these will be analysed at a later date and the data made available.

7.0 Mix Composition Analysis

A sub-sample was obtained from each of the mortar samples, with these prepared for determination of mix composition by acid digestion, with the aggregates recovered, following the acid digestion of the binder, for grading analysis. The results of the analysis carried out are presented in the following sections:

7.1 Mix Composition Analysis by Acid Digestion

Sample Ref.	**SR2600-S3**	**SR2600-S4**	
	15th *C* Mortar	**19th *C* Mortar**	
Binder Type	**Non-Hydraulic**	**non to Feebly Hydraulic**	
Lime : Aggregate Ratio	1.0 : 0.8	1.0 : 1.2	
Weight Proportions – Binder Type	***Quicklime***	***Quicklime***	***Hydrate***
Binder content	1.0	1.0	1.0
Fine Aggregate (Sand)	1.3	2.1	1.5
Volume Proportions			
Binder content	1.0	1.0	1.0
Fine Aggregate (Sand)	0.43	0.65	0.6

The mix proportions determined by this method includes all lime inclusions and any readily acid soluble aggregate as binder. Therefore, the results obtained tend to be lime rich, particularly where lime inclusions are present, and the binder content should be reduced by a factor to correct for this. To allow for this the mix composition has also been determined by modal analysis, see section 5 of report. The result of the grading analysis are presented in the table below and as aggregate filled histograms in the appended Figures No. 3 & 4.

Sample Ref:	**SR2600-S3**		**SR2600-S4**	
BS Sieve Size (mm)	**Retained %**	**Passing %**	**Retained %**	**Passing %**
8.00	0	100	0	100
4.00	0	100	1.8	98.2
2.00	1.2	98.8	0.9	97.3
1.00	4.5	94.3	2.2	95.1
0.500	26.0	68.3	9.7	85.4
0.250	42.5	25.8	38.0	47.4
0.125	13.7	12.1	37.5	9.9
0.063	5.5	6.6	5.2	4.7
Passing 63µm	6.6		4.7	

Table No. 7: Results of Grading analysis on recovered aggregates from mortar samples.

Based on the mineralogy of the sands and the particle size distribution it is likely that the two sands were obtained from different sources. The particles in S3 are sub-round to sub-angular with a degree of rounding, more than in sample S4. This along with the grading inferring sample S3 is more likely to be from the river Ure. Whereas, the aggregate in sample S4, which is more angular, is more typical of a pit sand source.

8.0 Summary

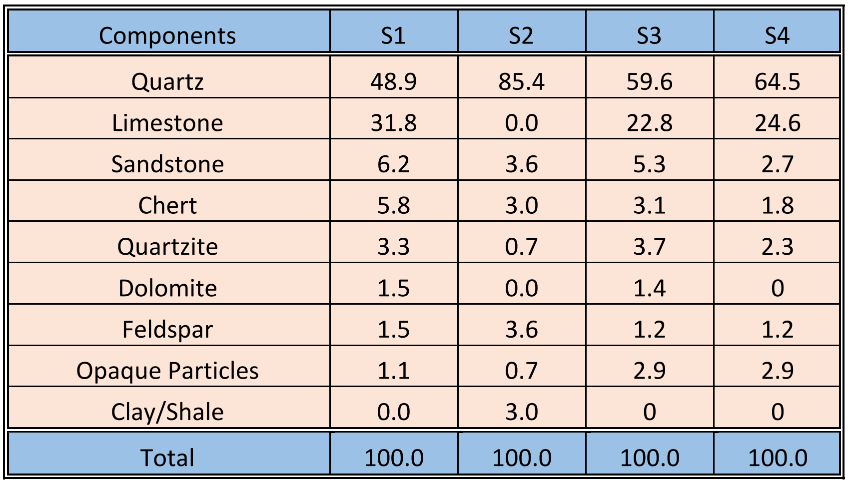
The mortars represented by the samples have been made from two different binders and this will most likely reflect the materials in use during the period that the works were executed, with these including both a Dolomitic lime, used in the form of a quicklime and a Magnesian/Dolomitic lime, dry slaked to a hydrate mixed with a pozzolan, possibly to impart a degree of hydraulicity to the mix. Details of the mix proportions are summarized below:

Sample Ref.	**SR2600-S3**	**SR2600-S4**	
Volume Proportions (by acid digestion – Total Binder content)			
Binder	***Quicklime***	***Quicklime***	***Hydrate***
Binder content (Quicklime)	1.0	1.0	1.0
Fine Aggregate (Sand)	1.3	2.1	1.5
Volume Proportions (by Chemical Analysis – Total Binder content)			
Binder content (Quicklime)	1.0	1.0	1.0
Fine Aggregate (Sand)	0.43	0.65	0.6
By modal analysis			
Binder: Agg. by vol. (Total)	1.0:0.62	1.0:1.7 + ash	
(Effective)	1.0:1.4	1.0:2.0 + 0.2 parts Ash	

Aggregates

As with the binder there is a difference between the aggregates used in each mortar. With that used in sample S3 being closer to the sands supplied from the River Ure than that from the Ouse. Whereas, the sand used in sample S4 was unlike either sand sample and, given its age, it is likely to have been supplied from another commercially available sand source. See results of particle size distribution and mineral proportions determined in the tables below:

Sample Ref:	**SR2600-S1**	**SR2600-S2**	**SR2600-S3**	**SR2600-S4**
Source	**River Ure**	**River Ouse**	**15th *C* Mortar**	**19th *C* Mortar**
BS Sieve Size (mm)	**Retained %**	**Retained %**	**Retained %**	**Retained %**
8.00	0	0	0	0
4.00	0	0	0	1.8
2.00	1.6	0	1.2	0.9
1.00	13.8	0	4.5	2.2
0.500	24.7	0.1	26.0	9.7
0.250	36.3	2.2	42.5	38.0
0.125	19.8	76.7	13.7	37.5
0.063	2.6	17.4	5.5	5.2
Passing 63µm	1.2	3.6	6.6	4.7



Components	S1	S2	S3	S4
Quartz	48.9	85.4	59.6	64.5
Limestone	31.8	0.0	22.8	24.6
Sandstone	6.2	3.6	5.3	2.7
Chert	5.8	3.0	3.1	1.8
Quartzite	3.3	0.7	3.7	2.3
Dolomite	1.5	0.0	1.4	0
Feldspar	1.5	3.6	1.2	1.2
Opaque Particles	1.1	0.7	2.9	2.9
Clay/Shale	0.0	3.0	0	0
Total	100.0	100.0	100.0	100.0

Tables 8 & 9: The above are provided for ease of comparing the aggregates in each of the samples submitted.

Limestone for lime production.

The Highmoor limestone supplied as a potential source of feedstock for the production of quicklime for use in restoration works, is a Dolomitic limestone, and as such is similar to that found as the binder in sample SR2600-S3 from the 15th century works. Whereas that used in the 19th Century works is more likely to have been a magnesian limestone, with a higher proportion of calcite.

On the basis of the calcining trials carried out in the laboratory, the limestone can be burned to produce a dolomitic lime. However, caution would need to be exercised when slaking the quicklime produced, as there may be a tendency for late slaking, if it is used and placed as a Hot Mixed Mortars (HMM).

Evidence of late slaking having occurred in the 15th Century mortars was observed in the thin section examination, with this also considered to be a possibility if the limestone is calcined for the same purpose, as indicated from the slaking of the quicklime in the laboratory. Where it was observed that the Lime slaked readily to Portlandite, but the Periclase only slaked to Brucite after several days soaking.

However, when the quicklime is incorporated within a larger mass of mortar, where the temperature would be raised significantly, over that achieved in the laboratory, and as long as there is sufficient water available, the rate of slaking would be expected to be much increased, as occurs when dolomitic quicklime is converted to a hydrate in modern day production, i.e. in an autoclave (with steam under pressure).

9.0 Quality Statement

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

The observations, analysis results, and comments offered herein, relate only to the samples of mortar received from the York Minster Fund, on the 29th May 2018. The mortar samples being from two ages of past construction, from the 15th and 19th centuries, along with samples of river sand from Rivers Ure and Ouse, in York, for comparison with the sands in the mortars. The sample of Highmoor Limestone was compared against that used originally in the samples received.

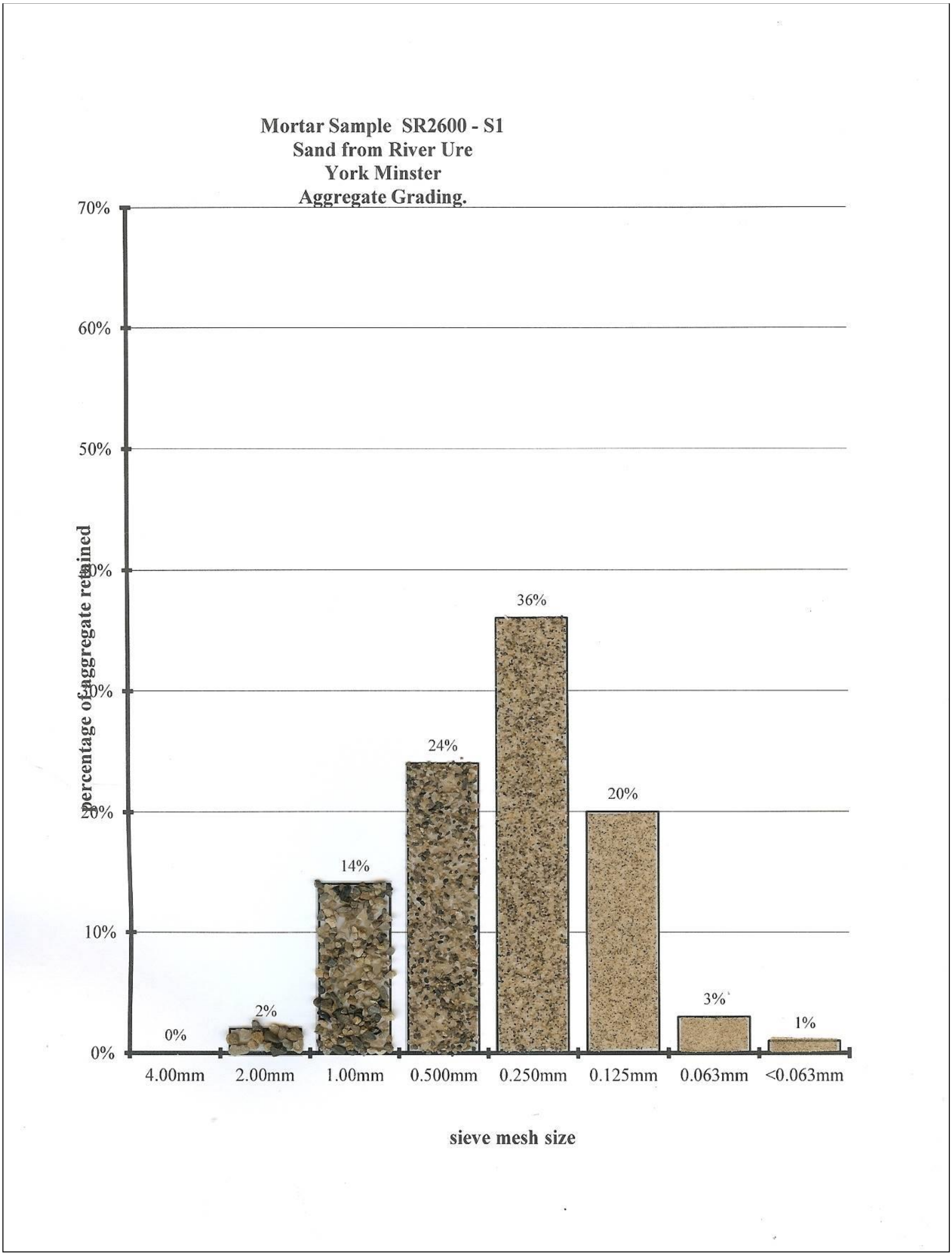


Figure No. 1: Grading of Aggregate from River Ure SR2600-S1

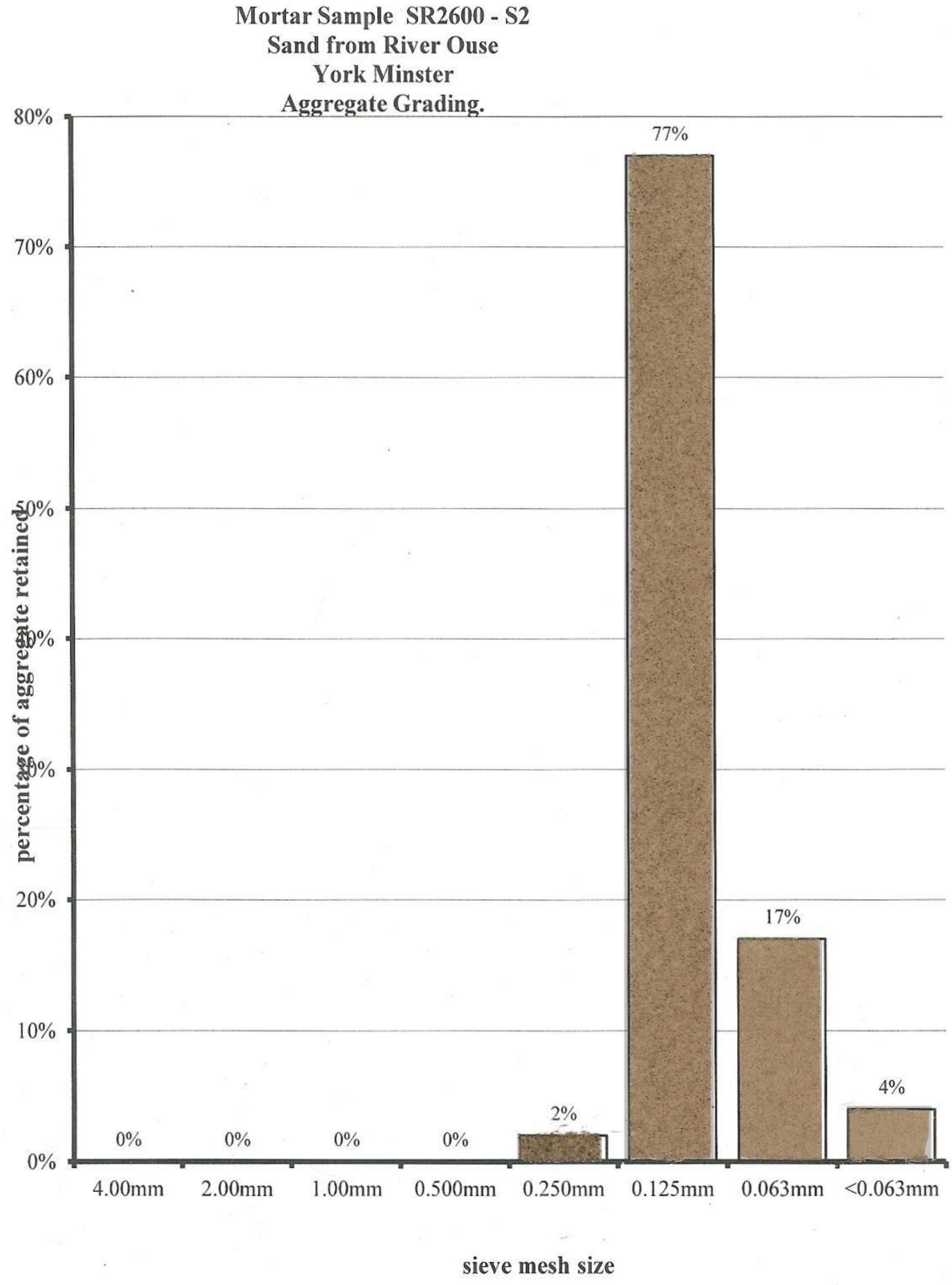


Figure No. 2: Grading of Aggregate from River Ouse SR2600-S2

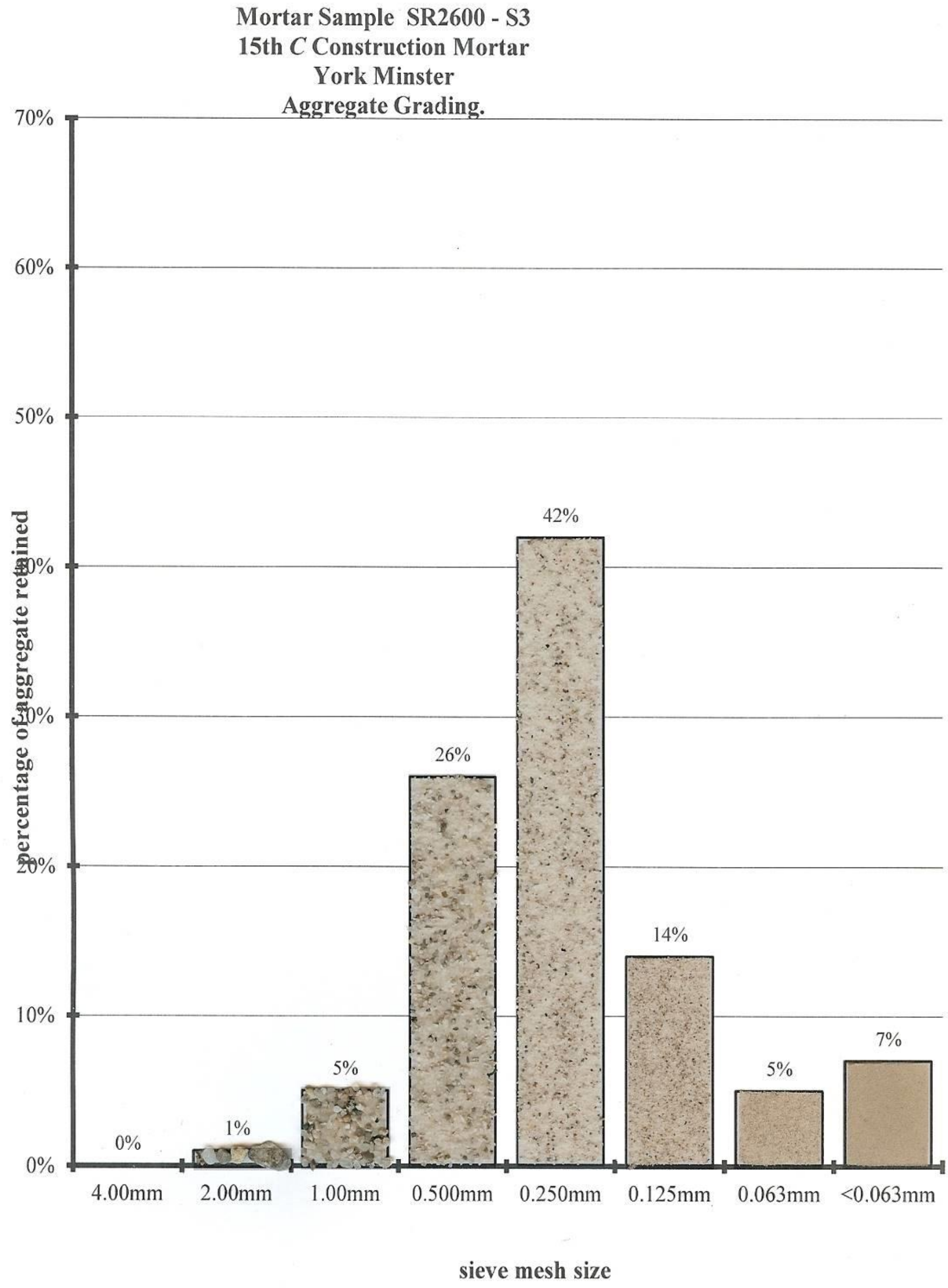


Figure No. 3: Grading of Aggregate from 15th Century Mortar

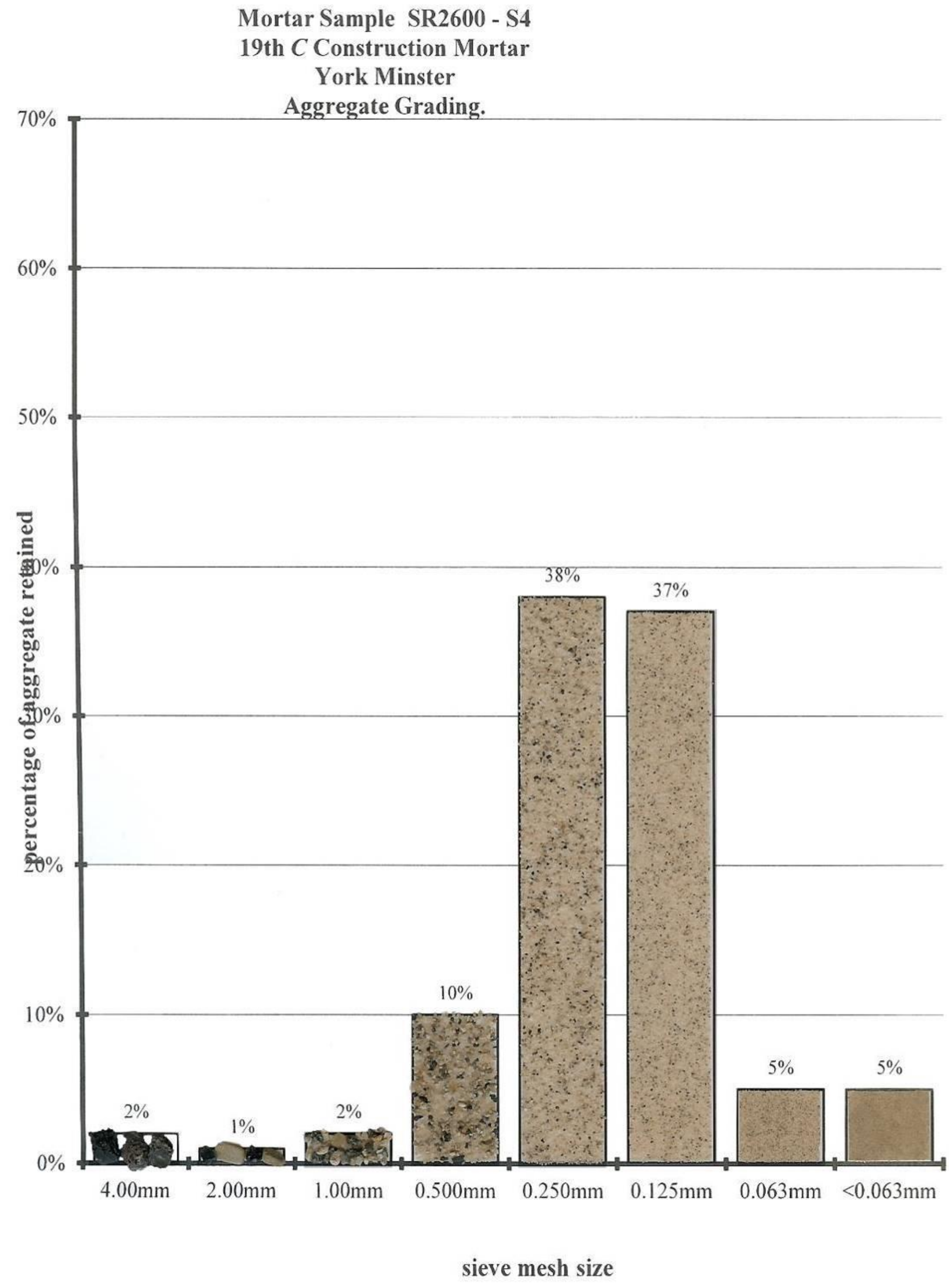


Figure No. 4: Grading of Aggregate from 19th Century Mortar SR2600-S4

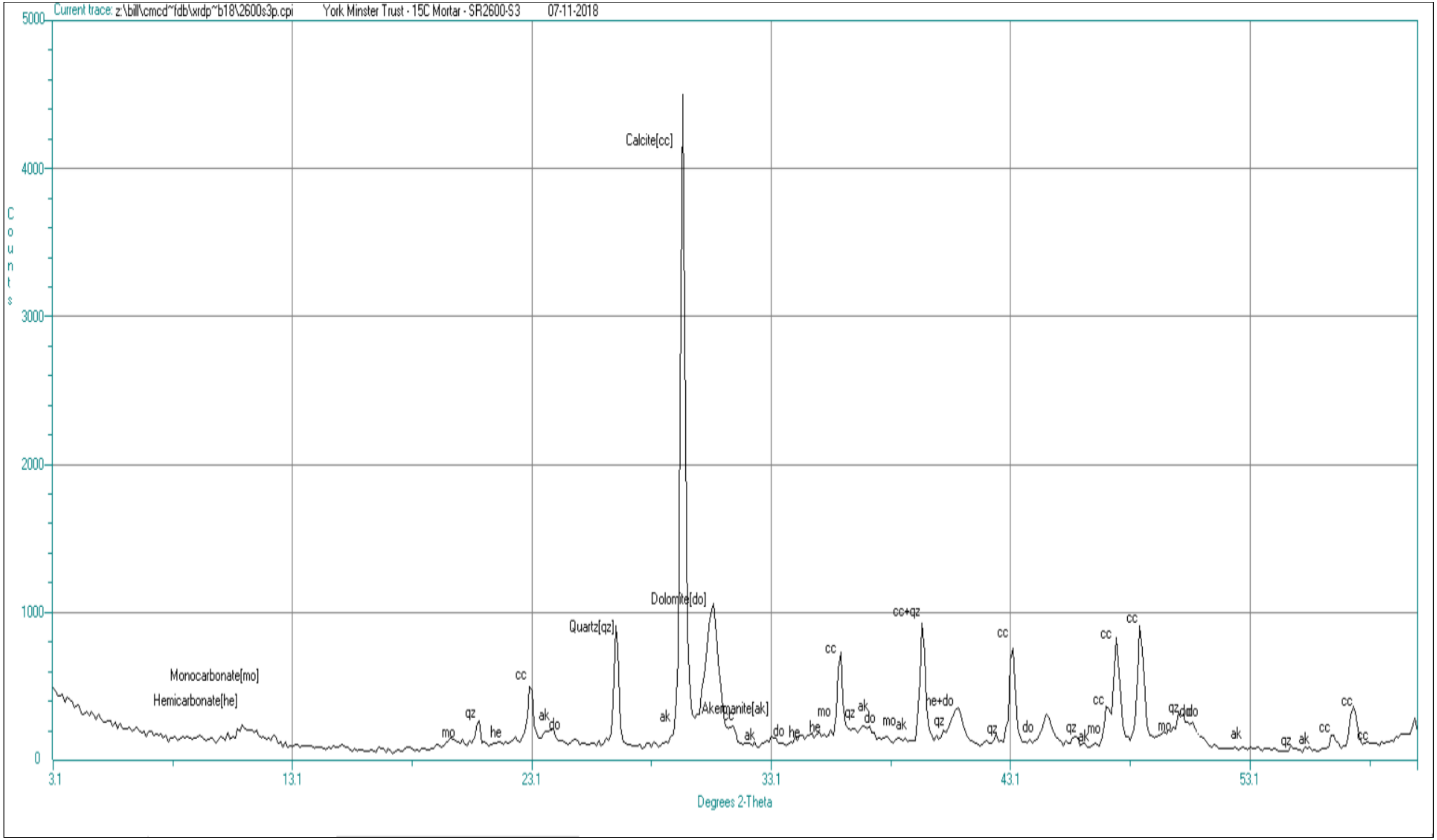


Figure No. 5 – SR2600-S3 – Binder from 15th Century Mortar ex York Minster.

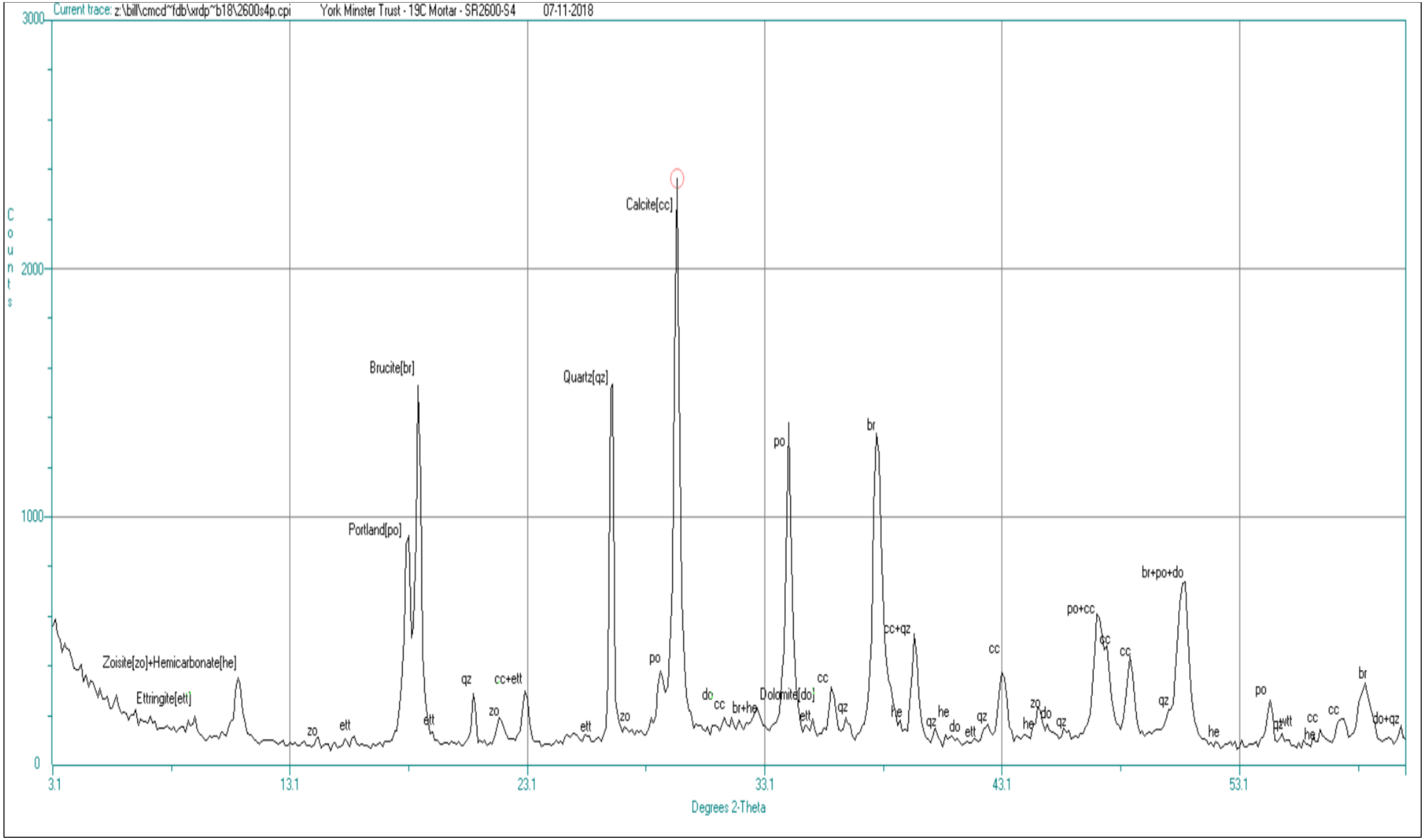


Figure No. 6 – SR2600-S4 – Binder from 19th Century Mortar ex York Minster.

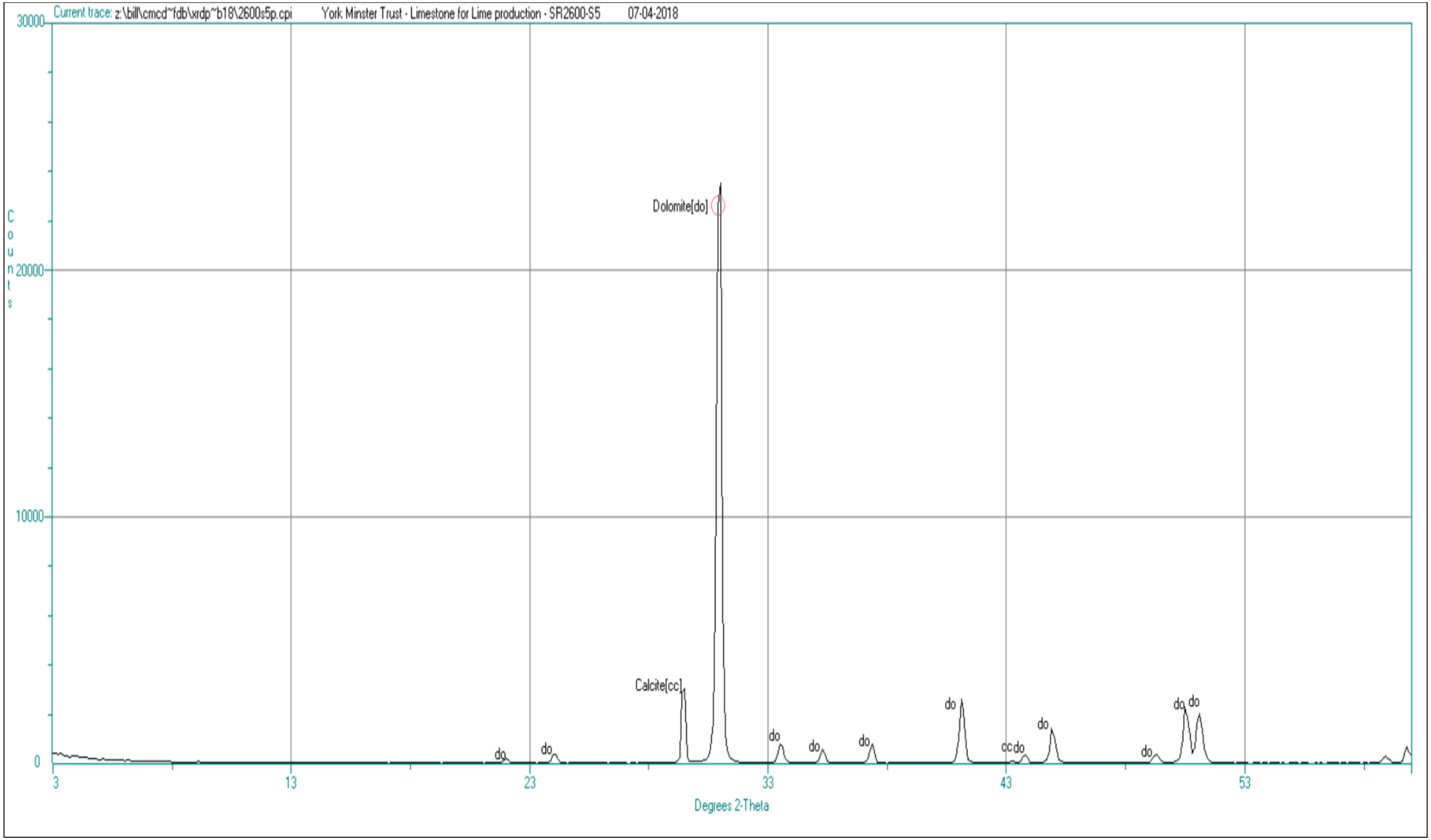


Figure No. 7 – SR2600-S5a – Highmoor Limestone, as received.

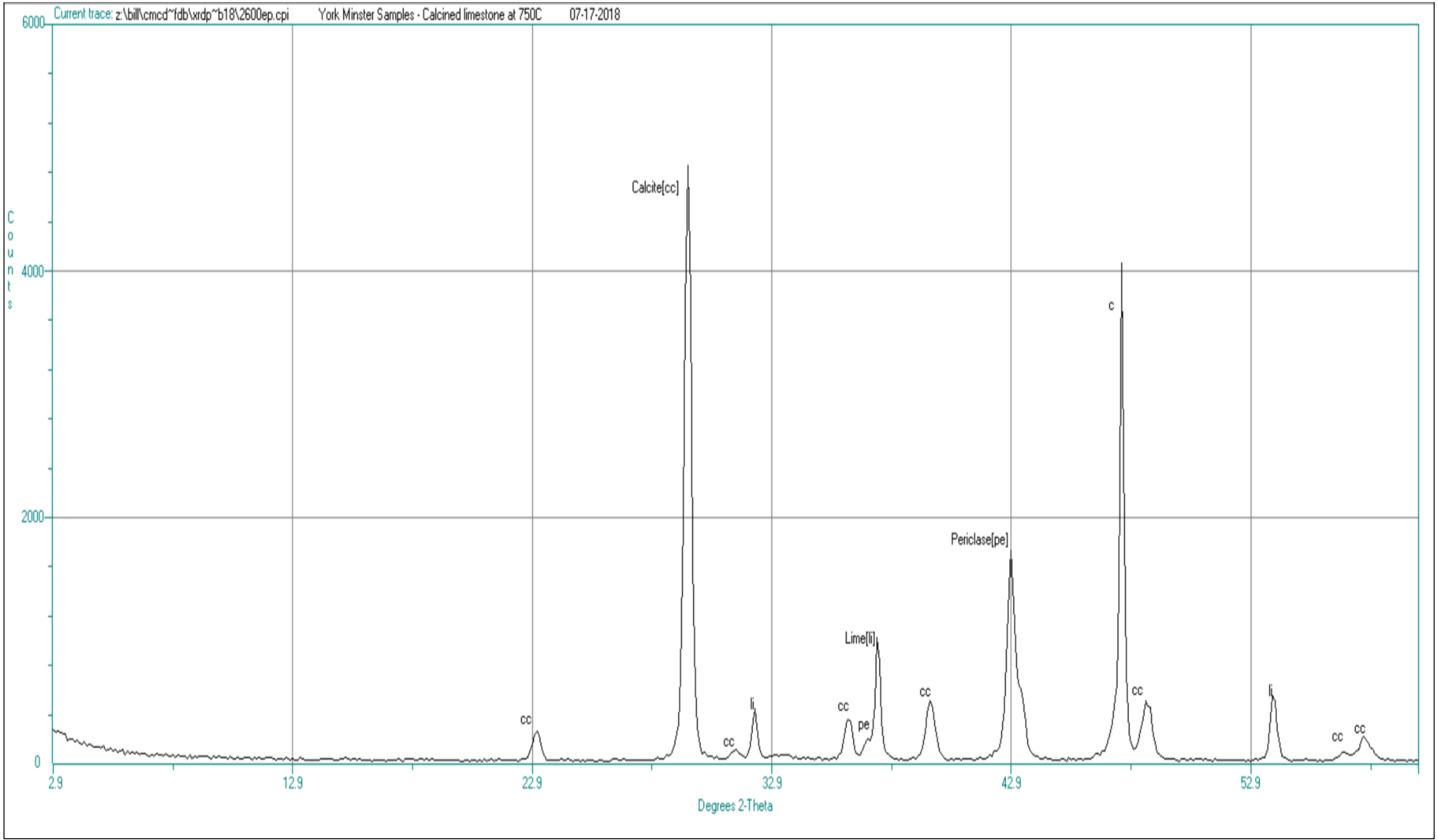


Figure No. 8 – SR2600-S5e – Highmoor Limestone, after calcining at 750°C.

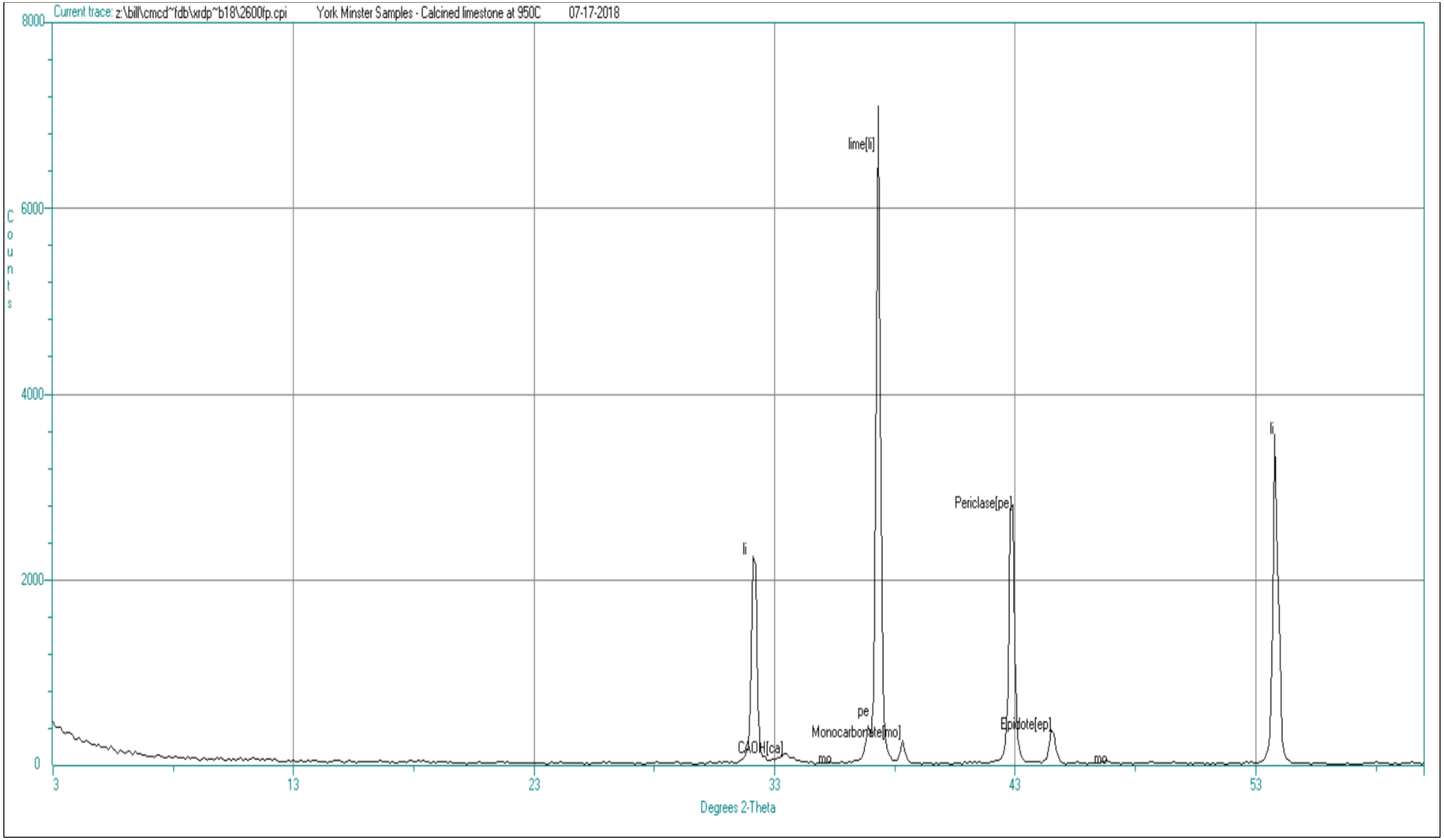


Figure No. 9 – SR2600-S5f – Highmoor Limestone after calcining at 950°C.

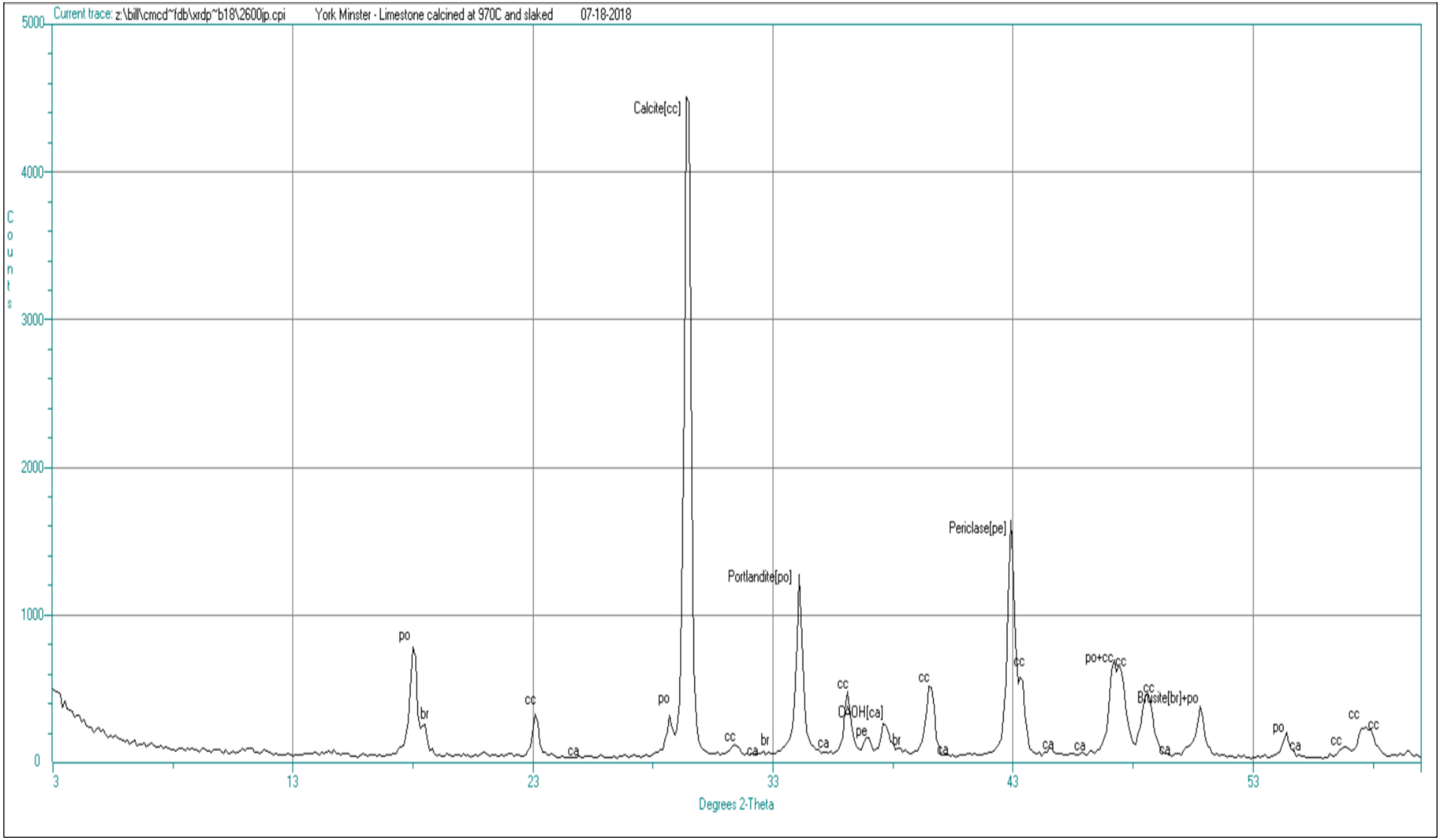


Figure No. 10 – SR2600-S5g – Highmoor Limestone, calcined at 750°C, after slaking.

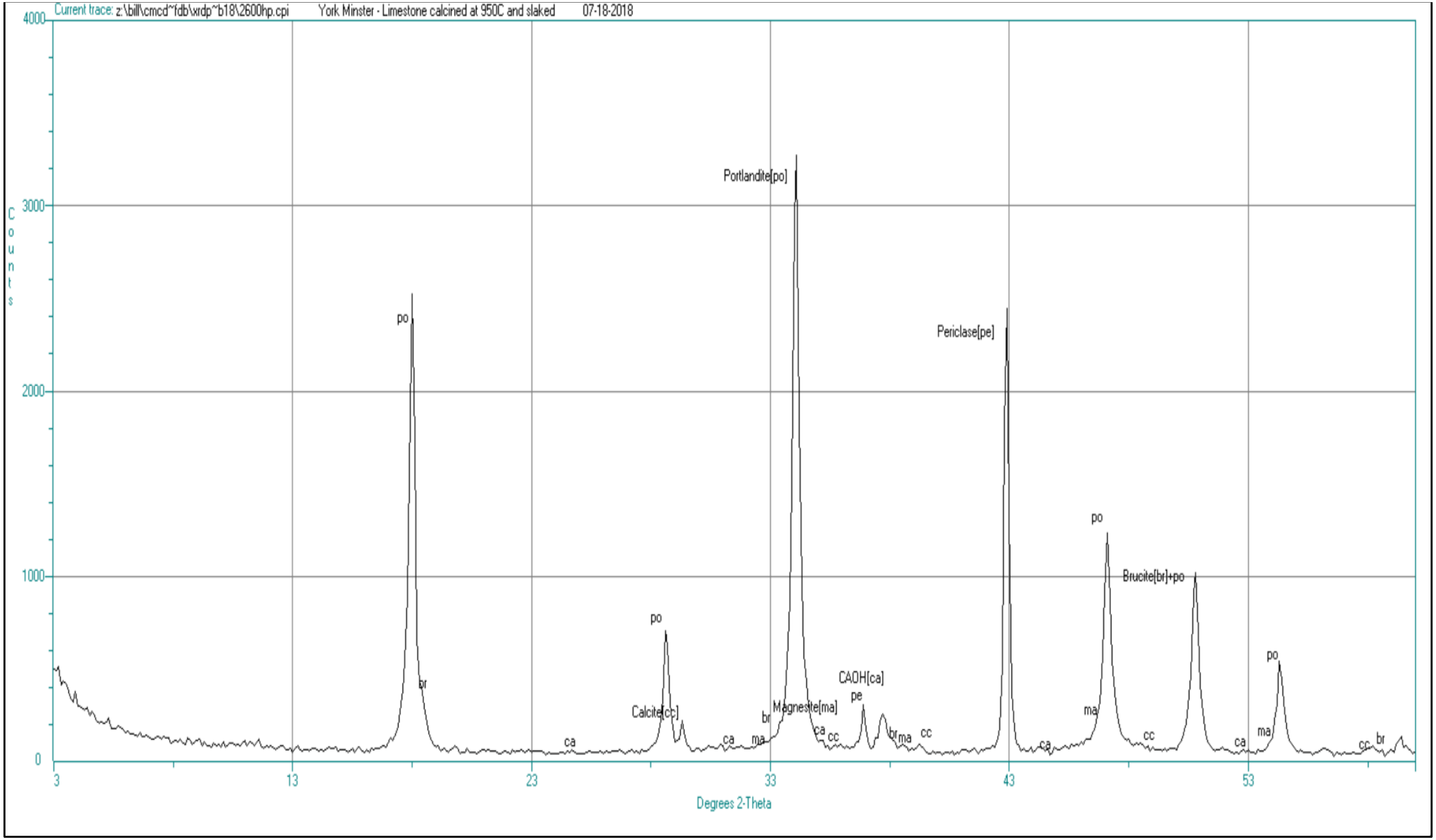


Figure No. 11 – SR2600-S5h – Highmoor Limestone, calcined at 950°C, after Slaking.

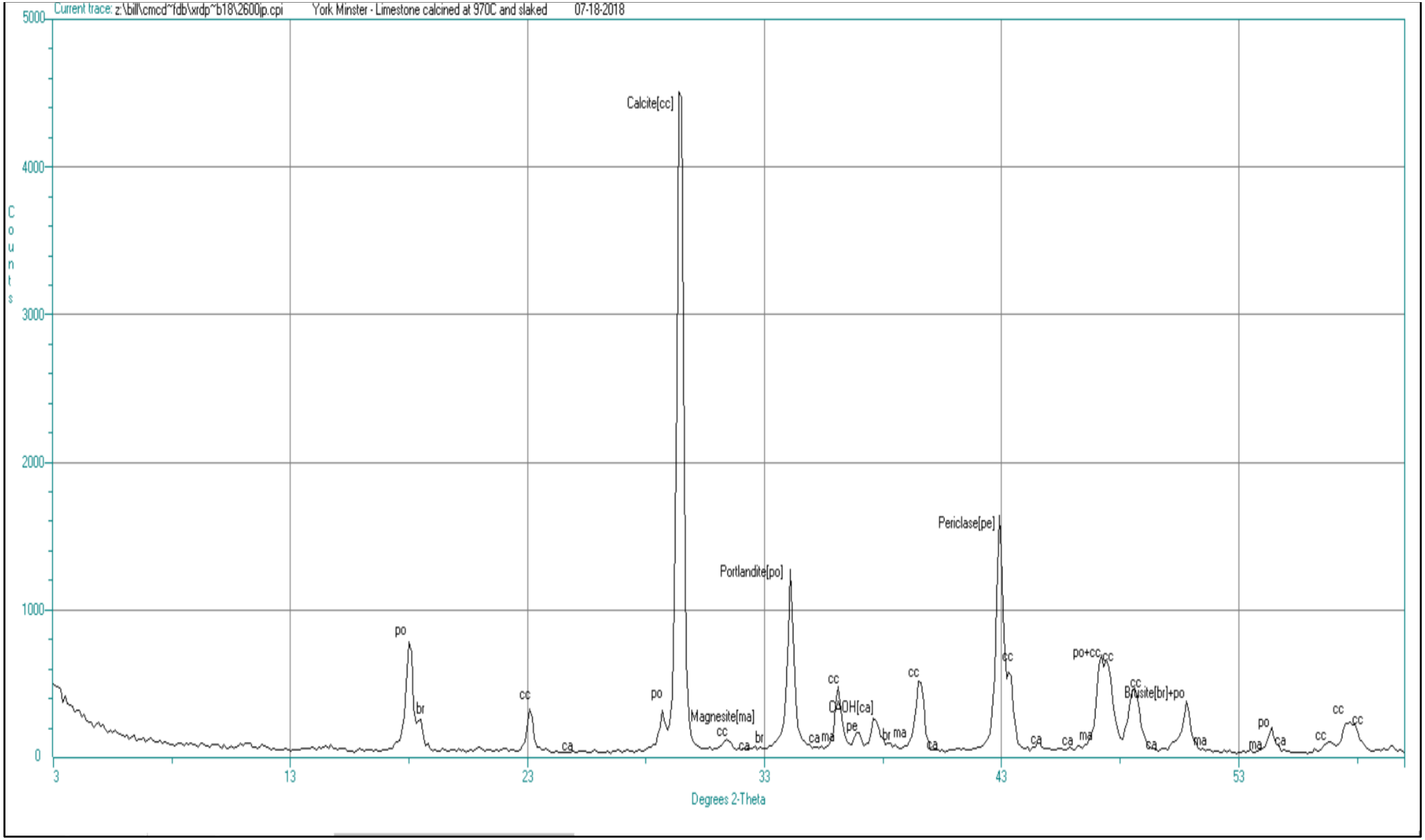


Figure No. 12 – SR2600-S5j – Highmoor Limestone, calcined at 950°C, after Slaking and 7 days carbonating.

APPENDIX "A"

Samples from a lime burn and mortar mix trial using Highmoor Limestone calcined in a field Kiln

Lime burn, and mortar mixed as an HMM by Nigel Copsey of the Earth, Stone & Lime Company, Pickering North Yorkshire.

1.0 Samples **Page 41**
2.0 Macroscopic Examination **Page 41**
3.0 Analysis by X-Ray Diffraction **Page 44**
4.0 Mix Composition Analysis by Acid Digestion **Page 45**
5.0 Properties of Mortar **Page 45**
Aggregate Filled Histogram **Figure A1**
X-Ray Diffractograms **Figures A2, A3, & A4**

1.0 Samples

A batch of samples was received in CMC's Stirling laboratory on the 2nd July 2018,
with the samples identified as materials from trial works associated with the York Minster Trust, were submitted by Nigel Copsey for examination and determination of the mortar properties.

On receipt in the laboratory the sample details were entered into the sample register and the unique sample reference SR2610 allocated. Details of the samples and type are detailed below along with the laboratory and Client sample references:

CMC Sample Reference.	Client Sample Reference.	Description/Comment
SR2610-S1	S1	Two lumps of well burnt limestone, Highmoor Limestone.
SR2610-S2	S2	Two lumps of over burnt limestone, Highmoor Limestone.
SR2610-S3	S3	A section of mortar, cast into a mould, made from sample S1.

A sample of Highmoor limestone was also submitted but as this was very similar to that supplied Previously, no further work was carried out on tis sample.

2.0 Macroscopic Examination

A summary of the properties of the samples, as received, is presented below:

CMC Sample No.	Client Ref	Mass (gram)	Maximum Dimension (mm)	Colour – Munsell Soil Colour Chart
SR2610-S1	S1	110.0	49.3 x 46.5 x 37.0	10YR 8/1 "Very Pale Brown" to 10YR 6/3 "Pale Brown".
SR2610-S2	S2	203.6	91.4 x 68.9 x 37.9	10YR 6/2 "Light Brownish Gey" to 10YR 5/1 "Grey"
SR2610-S3	Mortar	261.3	105.9 x 49.1 x 47.8	10YR 8/2 "Very Pale Brown"

Sample SR2610-S1 – Well Burnt Highmoor Limestone

Plate No. A1:

This sample consisted of two pieces of a well burnt limestone.

The quicklime broke easily on a light hammer tap and appeared to be burnt evenly throughout its thickness.

Sample SR2610-S2 – Overburnt Burnt Highmoor Limestone

Plate No. A2:

This sample consisted of two pieces of an overburnt limestone.

The quicklime required a moderate hammer impact to break and appeared to be overburnt throughout most of its thickness.

Sample SR2610-S3 – Mortar made from Highmoor Quicklime

Plates No. A3 & A4: The left plate shows the sample as received, with the right plate a freshly fractured surface treated with a phenolphthalein indicator solution showing that the mortar was uncarbonated.

The mortar was well compacted and moderately firm requiring firm finger pressure to break and disaggregate a piece sawn from the lump. It was advised that the sample was approximately 50 days old at date of test (cast on or around the 17th May 2018).

3.0 Analysis by X-Ray Diffraction

To assist in the identification of the composition of the binder in the mortar sample, a binder rich sub-sample was prepared and the mortar along with a sub-sample from each of the quicklime samples, with all three submitted to analysis by X-ray Powder Diffraction (XRD).

The prepared samples were analysed in a Philips X-ray Diffractometer run under the same operating conditions as for the initial York Minster samples. With the digital output from the diffractometer analysed in 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 obtained are shown in the following figures appended to the end of this Appendix, in the form of a labelled X-ray diffractograms:

Figure No. A2 – SR2610-S1 – Site calcined Highmoor Limestone, well burnt,
Figure No. A3 – SR2610-S2 – Site calcined Highmoor Limestone, overburnt,
Figure No. A4 – SR2610-S3 – Mortar made from Highmoor Limestone – HMM Trial.

The most abundant mineral components identified are indicated in the appended diffractograms using the following short-hand notation:

li = Lime (CaO) Dominant component from Calcining Calcareous limestone,
pe = Periclase (MgO) Magnesium Oxide, Dominant component from the calcining of Dolomitic Limestone,
po = Portlandite ($Ca(OH)_2$) Calcium Hydroxide, hydrated lime and from uncarbonated slaked lime binder,
br = Brucite ($Mg(OH)_2$) Magnesium Hydroxide, hydration product in a dolomitic lime,
cc = Calcite ($CaCO_3$) Calcium Carbonate, carbonated lime binder component, also the dominant component of any limestone in the aggregate,
va = Vaterite ($CaCO_3$) Calcium Carbonate, carbonated lime binder component, also found in some limestones in the aggregate and as reprecipitated lime,
do = Dolomite ($Ca,Mg(CO_3)_2$) Calcium Magnesium Carbonate, dominant component of Dolomitic limestone and in carbonated Dolomitic lime binders,
mo = Monocarbonate ($Ca_4Al_2O_6CO_311H_2O$) Calcium Aluminium Oxide Carbonate Hydrate, Carbonated *afm* phase, Hydration product in a hydraulic lime,
ps = Pseudibrookite ($TiO2$) Titanium Oxide, perhaps present as a contaminant in the limestone,
qz = Quartz (SiO_2) dominant component of the sand grains in the aggregates used in all of the mortar samples, and dominant mineral present in the sandstone sample.

In addition to the above, there are Feldspar minerals, including Microcline and Sanidine, along with trace proportions of clay minerals, these have not been identified in the diffractogram.

From the analysis it is indicated that the quicklime was burnt at temperatures close to the lab trials at 950°C, with Lime and Periclase being the dominant components.

The analysis of the mortar sample confirms that the lime readily slakes to Portlandite, but the Periclase is again slow to slake to Brucite, which, in the absence of lime inclusions should not give rise to problems relating to unsoundness, however, if used to build large sections, it may be prone to a degree of expansion as the lime hydrates in place.

If the mortar is to be mixed as an HMM it is perhaps advisable to dry slake the mix, screen out any oversize and over/under burnt lime and incompletely slaked quicklime, prior to re-tempering the mortar and placing it cold. If the mortar can be stored for a period, to encourage complete conversion of the Periclase to Brucite, the less risk there will be of any unsoundness in the mortar.

Proportions by Rietveld Refinement.

Material:	**Highmoor Limestone Quicklime**		**Mortar sample**
	Calcined well	**Over burnt**	
Sample Ref:	**SR2610 – S1**	**SR2610 – S2**	**SR2610 – S3**
		% by Mass	
Lime	48.4	52.7	-
Periclase	44.7	44.2	25.4
Portlandite	3.2	2.3	16.3
Brucite	1.2	0.8	0.8
Calcite	0.5	-	43.4
Vaterite	-	-	0.6
Dolomite	-	-	4.9
Pseudobrokite	2.0	-	-
Quartz	-	-	8.1
Microcline (Feldspar)	-	-	0.4
Sanidine (Feldspar)	-	-	0.1

4.0 Mix Composition Analysis by Acid Digestion

Sample Ref.	**SR2610-S3**
Binder Type	**Non to Feebly Hydraulic**
Lime : Aggregate Ratio	1.0 : 2.6
Weight Proportions – Binder Type Quicklime	
Binder content	1.0
Fine Aggregate (Sand)	4.7
Volume Proportions	
Binder content	1.0
Fine Aggregate (Sand)	1.5

As it is normal for mix proportions determined by this method to include all lime inclusions and any readily acid soluble aggregate, as binder. Therefore, the results obtained tend to be lime rich. To minimise the risk of the dissolution of any limestone aggregate the acid used was diluted to 5% with the dwell time minimised, but still sufficient to digest all of the binder.

The result of the grading analysis carried out on the recovered aggregate, are presented in the table below and as an aggregate filled histogram, see appended Figure No. A1.

Sample Ref: SR2610-S3

BS Sieve Size (mm)	Retained %	Passing %
8.00	0	100
4.00	2.9	97.1
2.00	17.4	79.7
1.00	19.8	59.9
0.500	23.9	36.0
0.250	19.1	16.9
0.125	11.2	5.7
0.063	3.7	2.0
Passing 63µm	2.0	

5.0 Properties of the Mortar

As a request for a measure of the mortar strength was received with the sample, and as the sample was of sufficient size, two small prisms were sawn from the block received, and these prepared for test.

The prisms were measured, and their density determined prior to them being capped with plaster of Paris for determination of their compressive strength.

The results obtained are presented below:

Property	Mean value
Bulk Density	1625kg/m³
Compressive Strength	0.3Mpa

The mortar was tested at approximately 50 days after casting with the mortar mostly uncarbonated, with only surface carbonation identified from a phenolphthalein indicator test carried out on the prisms after test.

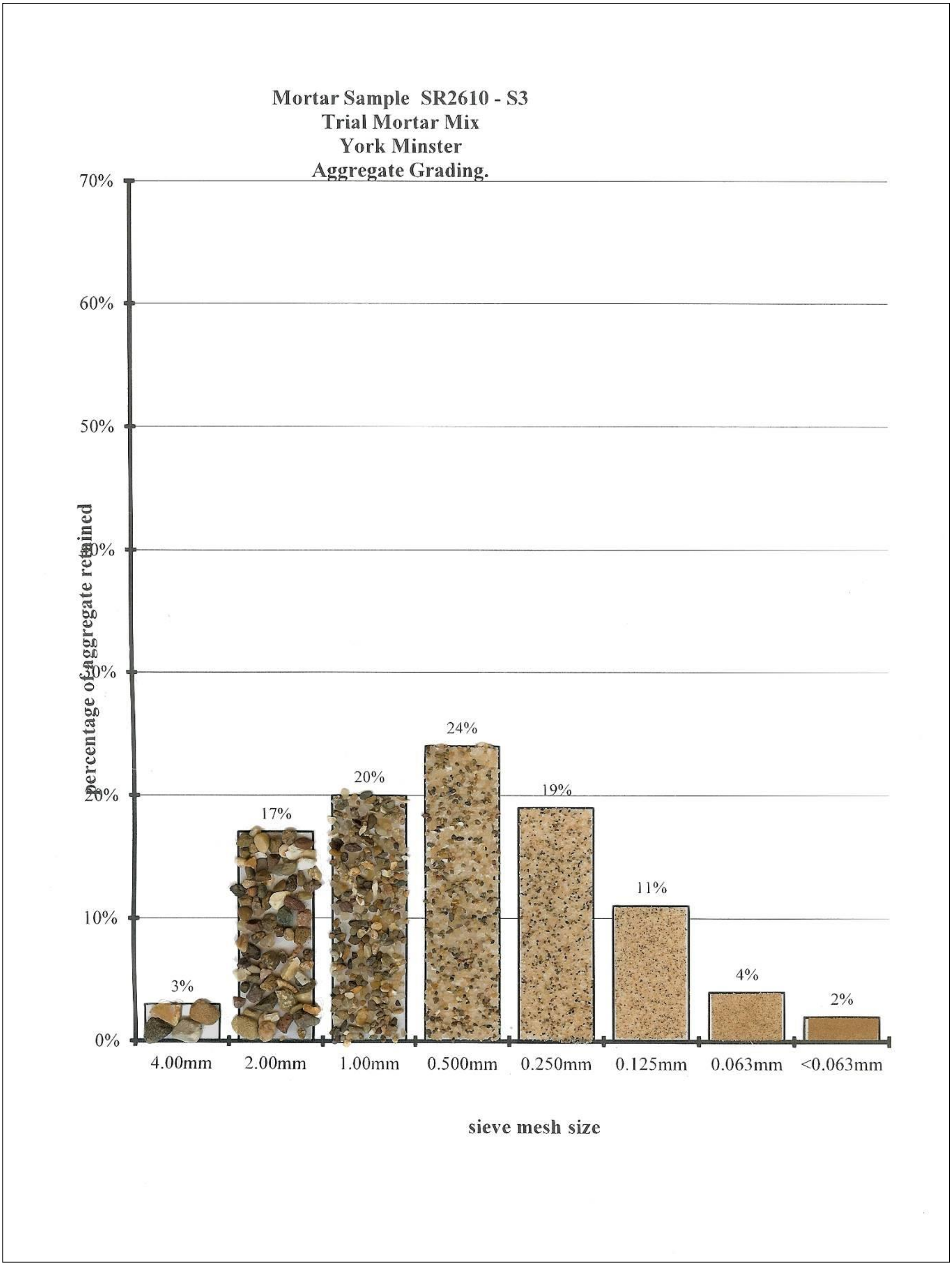


Figure No. A1: Grading of Aggregate from mortar sample SR2610-S3

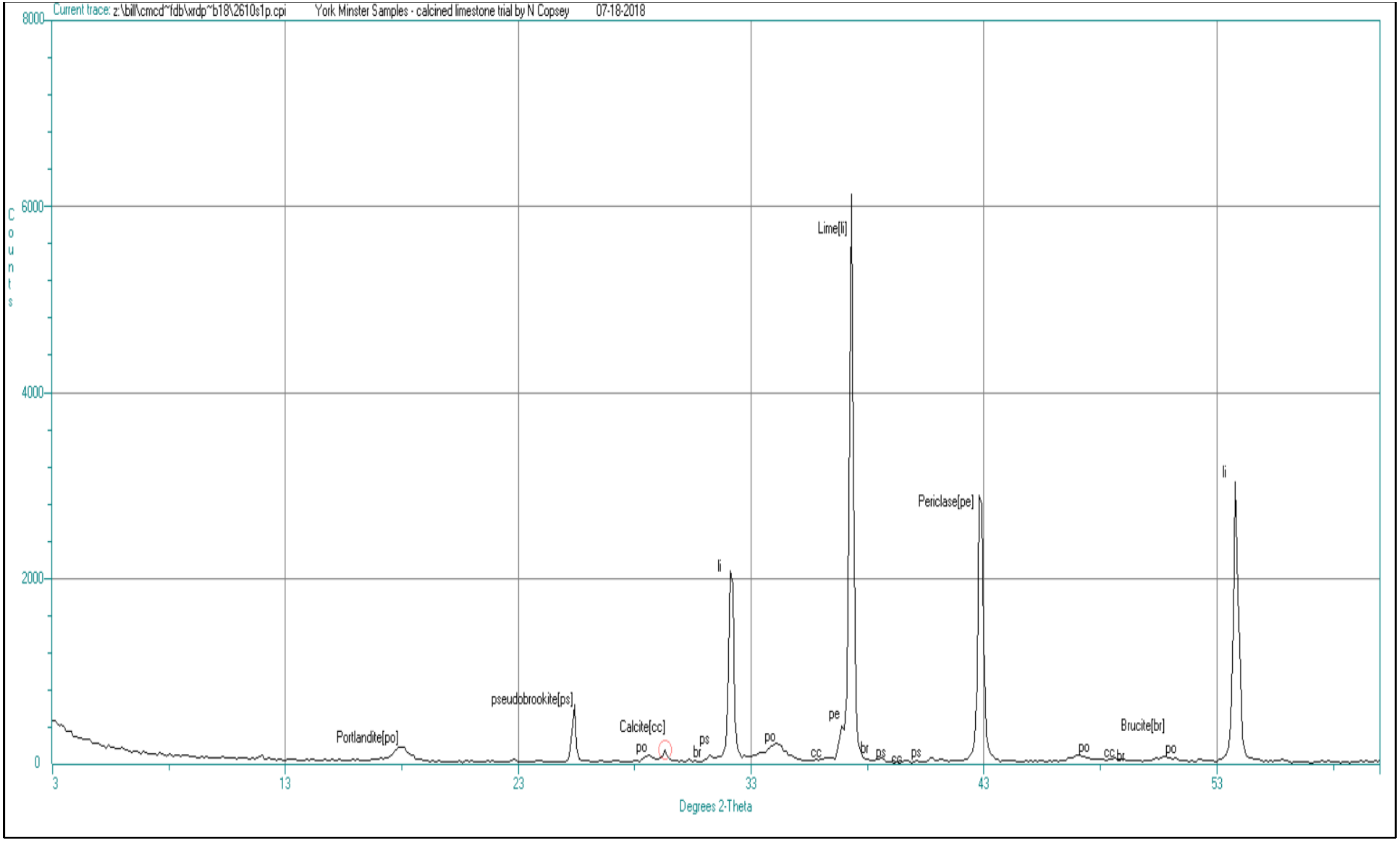


Figure No. A2 – SR2610-S1 – Site calcined Highmoor Limestone, well burnt.

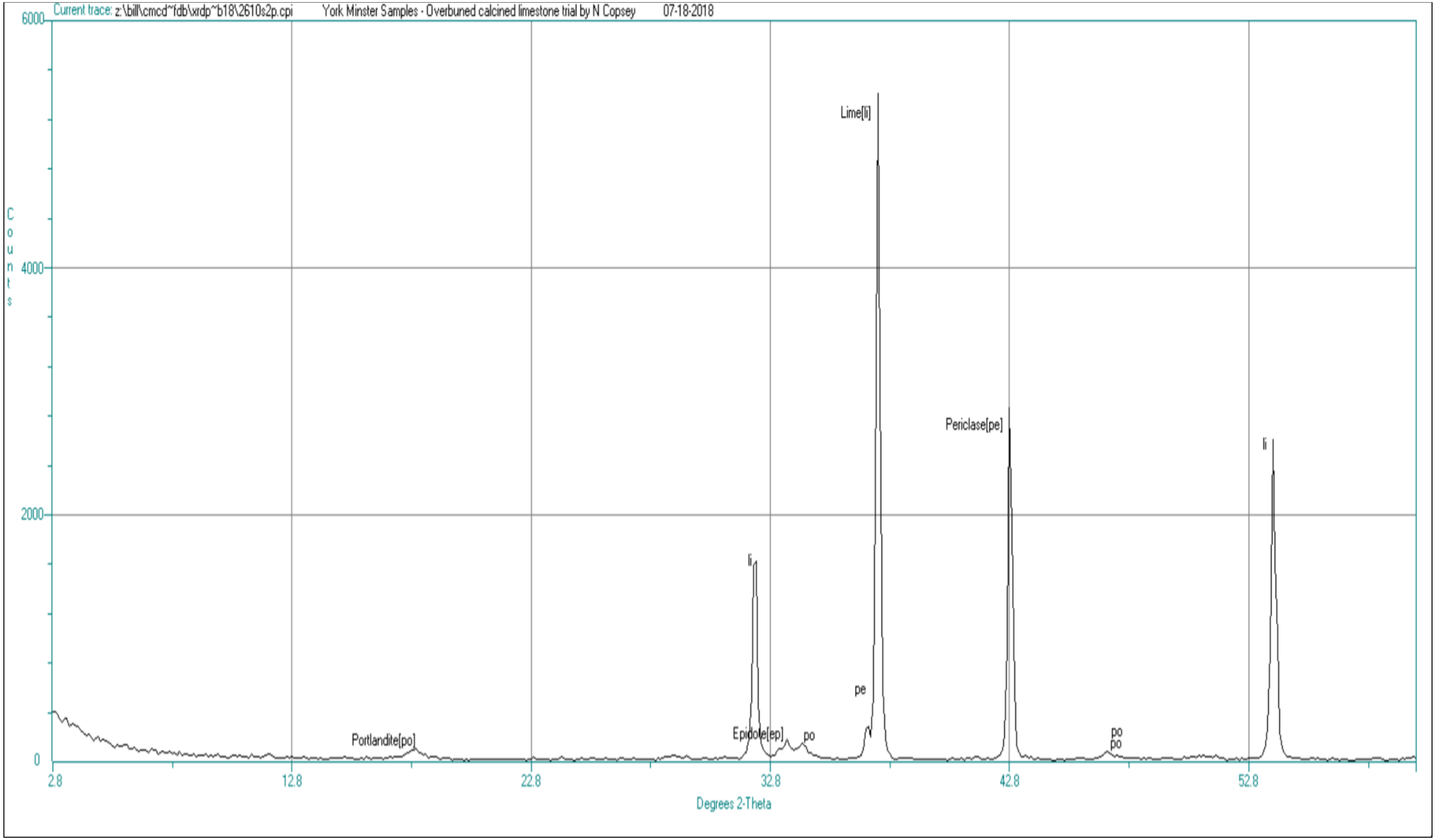


Figure No. A3 – SR2610-S2 – Site calcined Highmoor Limestone, overburnt.

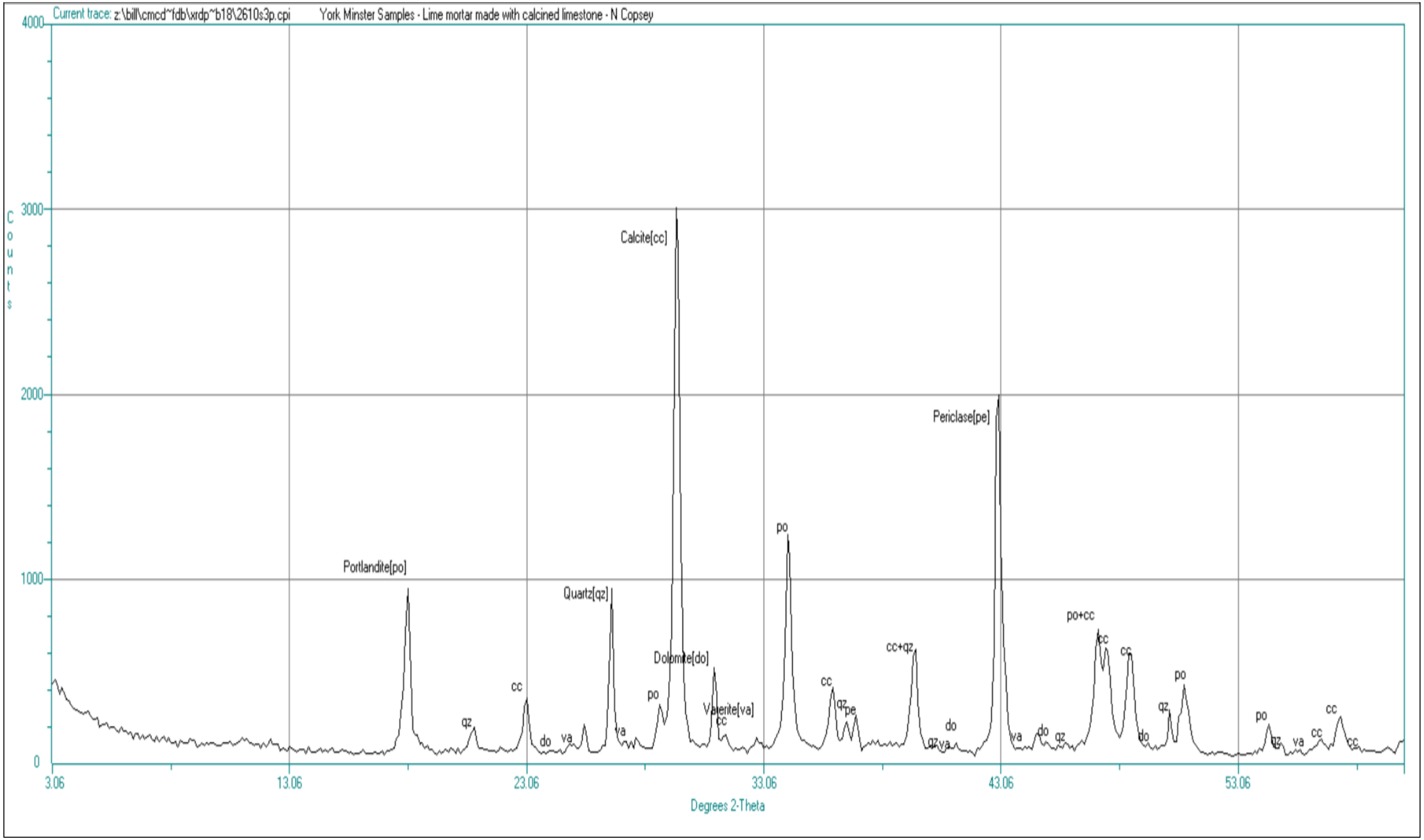


Figure No. A4 – SR2610-S3 – Mortar made from Highmoor Limestone – HMM Trial.