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Our Ref: M/1991/19/C4
Your Ref.: T2019

4th June 2019

CERTIFICATE OF ANALYSIS OF MORTAR SAMPLES FOR DETERMINATION OF MIX COMPOSITION & BINDER TYPE

Project Reference	:	North Yorkshire Moors – This Exploited Land of Iron
Sample Location	:	Rosedale East Stone Kilns
Sample Description	:	RESK1A – Typical Vault Brickwork, Kiln 1 Vault 4 RESK1B – Typical Vault Brickwork, Kiln 4 Vault 13 RESK2 – Rear Retaining, Wall kiln 4 RESK3 – Front Wall/Facing, kiln 4, Bedding mortar
Date Received	:	12th March 2019
CMC Sample Ref	:	SR 2706-S1A = RESK1A – Kiln 4, Vault 4, Brick mortar SR 2706-S1B = RESK1B – Kiln 4, Vault 13, Brick mortar SR 2706-S2 = RESK2 – Kiln 4, Rear Retaining Wall SR 2706-S3 = RESK3 – Kiln 4 Front Wall/Facing
Date Analysed	:	5th April to 20th May 2019
Method of Test	:	Determination of binder type by X-Ray Diffraction analysis. Mix composition by acid digestion with grading analysis of recovered aggregate, and thin section examination.

Sample

Four individual samples of mortar were received from the Rosedale East Stone Kilns, as one of the five batches of mortar samples received in CMC's Stirling Laboratory on the 12th March 2019. The samples were received from Structural and Civil Consultants Ltd., Northallerton, on behalf of the North York Moors National Parks Authority as part of their "This Exploited Land of Iron" project. The samples were identified as having been obtained from the Kiln masonry at Rosedale East Stone Kilns, in the North Yorkshire Moors.

The samples were to be submitted to a selection of analyses in an attempt to determine the composition of the mortar in each sample, with the grading of the aggregates recovered from the mortars. In addition, comment was also requested on the condition of the mortars as received, and, if possible, on the type of binder used in the mortar production, and the form in which it was used.

On receipt in the laboratory, the sample details were entered the sample register and the unique sample identification number SR2706 allocated.

Details of the individual samples submitted for examination and analysis given below:

CMC Sample Ref.	Client Ref	Location Sampled
SR2706 – S1A	RESK1A	Typical Vault Brickwork, Kiln 1 Vault 4,
SR2706 – S1B	RESK1B	Typical Vault Brickwork, Kiln 4 Vault 13
SR2706 – S2	RESK2	Mortar from Rear Retaining Wall, Kiln 4
SR2706 – S3	RESK3	Mortar from Front Wall/Facing, Kiln 4.

Method of Test

On receipt in the laboratory the samples were logged, with their mass and size recorded prior to being photographed, in the as-received condition. All samples were 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 samples were exposed to a series of *ad hoc* droplet tests employing a range of reagents and indicator solutions both to assist in the identification of the components present and to assess the condition of the mortars as received.

Following the initial examination binder rich sub-samples were obtained from each sample, and variant present, for X-ray Diffraction (XRD) analysis. This technique was employed to permit identification of the binder type used in the production of the mortars, as it would also clarify if there were any crystalline contaminants or reaction products present.

On the basis of the results from the XRD analysis, the samples in which the binders were indicated to be non to feebly hydraulic were prepared for mix composition by acid digestion, following the methods of the Scottish Lime Centre Trust (SLCT). With the insoluble residue recovered, following the acid digestion, washed dried and sieved through a nest of British Standard sieves to permit the grading of the aggregate to be determined.

On the basis that a proportion of the aggregate may consist of limestone, or other acid soluble components, a petrographic thin section was prepared from each sample. In addition to clarifying the aggregate types present, this would permit clarification of the form in which the binder was used at the time of production, along with providing confirmation of the mix composition by modal analysis, thereby permitting a correction for the presence of any acid soluble aggregate components, and isolated lime inclusions, present in the mix.

Observations from a Macro/Microscopic examination

On receipt in the laboratory the sample were logged with the following determined:

Sample Ref.	Client Ref.	Mass of Sample (gram)	Dimensions of Largest piece (mm)	Colour by the Munsell Soil Colour Charts
SR2706-S1A	RESK1A	20.8	48.7 x 26.0 x 12.1	10YR 8/2 “Pinkish White”
SR2706-S1B	RESK1B	89.4	54.1 x 41.8 x 27.7	10YR 8/1 “White”
SR2706-S2	RESK2	156.7	49.5 x 38.9 x 28.9	10R 8/3 “Pink”
SR2706-S3	RESK3	74.2	61.7 x 39.2 x 20.9	10R 8/1 “White”

Sample SR2706-S1A (RESK1A): Typical Vault Brickwork Mortar – Kiln 4 Vault 1 – Pinkish White

This sample consisted of four fragments of a pinkish coloured mortar along with a small quantity of fines. The mortar fragments were well compacted and moderately hard and strong. However, the intact pieces could be broken under persistent firm finger pressure, with the pieces breaking with an audible 'snap', indicating a degree of brittleness in mortar. The freshly fractured surfaces were found not to be friable.

On examination the mortar was noted to contain an abundance of lime inclusions, up to 3.5mm in size. The inclusions were observed to be mostly sub-angular in shape, with a few retaining harder cores, perhaps indicating incompletely calcined lime.

Plates No. 1 & 2: The left plate shows the intact pieces in this part of the mortar sample. With the right plate showing a magnified image of a freshly fractured surface through the thickness of the sample. Note the abundance of lime inclusions, and possible limestone, observed randomly distributed throughout.

On testing a freshly fractured surface with phenolphthalein indicator the mortar was found to be fully carbonated. With water droplet tests confirming that the mortar was porous with the droplets being rapidly absorbed into the fabric.

The aggregates appear to be dominated by dark minerals and limestone, with quartz in the finer fractions along with a minor ash proportion. The coarser grains are a mixture of limestone, partially burnt and unburnt, with coal, coal ash clinker and brick fragments. The lime fragments measured up to 4.0mm in size with the clinker and coal up to 1mm in size and the weathered brick fragments are up to 0.2mm in size, noted along with a proportion of coal ash. The aggregates are sub-angular to sub-round and elongate in shape.

Sample SR2706-S1B (RESK1B): Typical Vault Brickwork Mortar – Kiln 4 Vault 13 - White Mortar

This sample contained ten intact pieces of mortar along with a small quantity of fines. The larger fragments were well compacted and moderately hard to hard, requiring firm finger pressure to break. The mortar in all fragments were noted to contain lime inclusions distributed throughout, with the inclusions being angular to sub-round in shape and measuring up to 4.8mm in size.

On testing several freshly fractured surfaces with a phenolphthalein indicator the mortar fragments tested were all found to be carbonated throughout their thickness.

The water droplet tests showed that the droplets were rapidly absorbed and diffused throughout the fabric of the mortar, to depth, indicating a well-connected pore structure and high porosity.

Plates No. 3 & 4: The left plate shows the sample as-received. The right plate shows a freshly fractured surface through the thickness of a large fragment, in which an abundance of lime inclusions and limestone fragments were apparent, with small dark coal fragments and red brick fragments distributed throughout.

Entrapped air voids measured up to 1.3mm in size were observed, with these containing an abundance of needle-like crystals (sulphate minerals) along with a patchy crystalline calcite coating, which would infer water percolation, through the mortar over time.

The aggregates are sub-angular to irregular to elongate in shape and are dominated by limestone fragments, coal, clinker and ash along with a low proportion of fine quartz grains and other lithic fragments.

Sample SR2706-S2 (RESK2): Rear Retaining Wall, Kiln 4 – Pink Mortar

This sample consisted of an abundance of fragments, with a variable composition, perhaps indicating more than one mortar type in the sample, or, more likely, that the mortar had been poorly mixed.

Plates No. 5 & 6: The left plate shows the sample, as received. The right plate shows a close-up of a freshly fractured surface, where the dense condition of the fabric can be seen along with abundance of angular white lime inclusions, grey and white limestone fragments, dark coal fragments, with patches of grey ash.

The mortar fragments were very well compacted, hard to moderately hard, with a dense fabric. The fragment broke under persistent firm finger pressure, and once broken could be powdered under firm finger pressure.

On testing freshly fractured surfaces with a phenolphthalein indicator the mortar was found to be fully carbonated, and from placing water droplets onto freshly fractured surfaces, these were quickly absorbed, again confirming the presence of a well-connected pore structure within the thickness of the mortar.

The aggregates are angular to irregular and elongates in shape and are dominated by limestone fragments, with coal, coal clinker and ash, with minor brick fragments and a very low proportion of natural sand grains, containing quartz and indeterminate lithic fragments.

Sample SR2706-S3 (RESK3): Front Wall/Facing, Kiln 4, Bedding Mortar – White Mortar

This sample consisted of four fragments of mortar and a very small quantity of fines. The mortar fragment were all well compacted, and were very hard and strong, however, they could be broken under persistent firm finger pressure, and once broken the fractured surfaces became friable.

Plates No. 7 & 8: The left plate shows the four intact mortar fragments in the sample as received. The right plate shows a close-up of a fractured surface, through the largest intact piece. This shows a dense fabric, along with an abundance of lime inclusions and limestone fragments.

Phenolphthalein indicator tests confirmed that the mortar was fully carbonated. With water droplet tests indicating that droplets placed onto soiled outer surfaces were supported for an extended period, with those applied to fractured surfaces being quickly absorbed. This would infer that the mortar had a well-connected internal pore structure. With the outer surfaces coated in redeposited calcite with entrapped soiling, which had the effect of blocking the pore structure and sealing of the outer surfaces.

On examination of the fractured surfaces an abundance of white lime inclusions were observed, with these measuring up to 7.2mm in size. The inclusions are angular to sub-round in shape and have the appearance of being formed from a mixture of quicklime and limestone fragments, the latter including partially over burnt particles along with fresh, unburnt, limestone.

The aggregates are angular to irregular and locally elongated in shape. These include dark minerals that are composed of coal, coal clinker, brick fragments along with a minor proportion of quartz and other indeterminate lithic fragments. The aggregates had a maximum size of 7.5mm but were mostly finer than 0.5mm.

Results of XRD Analysis for Binder Type

To help clarify the mineral and crystalline components present in the mortar samples received, a representative sub-sample was obtained from each mortar sample, and each variant observed to be present, with these crushed and lightly ground in an agate mortar and pestle in preparation for analysis. During grinding care was taken to minimise the crushing of the aggregate particles, as if present in abundance, in the analysis sample, they could mask any hydraulic components present, which may only be present in trace proportions.

The ground and powdered materials were sieved over a 63µm sieve to remove as much of the aggregate component as possible, thereby permitting a binder rich sub-sample to be obtained for analysis. The prepared powdered samples were back-packed into proprietary sample holders for presentation in the diffractometer, with this technique employed to ensure, as close as possible, the true random orientation of the components present.

All samples were analysed in a Diffractometer which 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. With the digital output analysed by a computer program, which matched the peak positions against the JCPDS International Standard Mineral Data-base sub files using a search window of 0.1°.

The results obtained from the analysis are presented in the following attached Figures, in the form of labelled X-ray Diffractograms:

Figure No. 4: SR2706-S1A (RESK1A) Kiln 1, Vault 4, Brickwork, Pinkish White mortar,

Figure No. 5: SR2706-S1B (RESK1B) Kiln 4, Vault 13, Brickwork, White mortar,

Figure No. 6: SR2706-S2A (RESK2A) Kiln 4, Rear Retaining Wall, Friable Pink mortar,

Figure No. 7: SR2706-S2B (RESK2B) Kiln 4, Rear Retaining Wall, Hard Pink mortar,

Figure No. 8: SR2706-S3 (RESK3) Kiln 4, Front Wall/Facing, White bedding mortar.

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

- **cc** = Calcite ($CaCO_3$) Calcium Carbonate, carbonated lime from lime binder and any limestone aggregate present in the mortar,
- **ar** = Aragonite ($CaCO_3$) another crystalline form of Calcium Carbonate, from limestone, commonly associated with shell and found in some forms of redeposited leached lime binder,
- **qz** = Quartz (SiO_2) Silicon Oxide, a component of the aggregate in some of the mortars,
- **he** = Hematite (Fe_2O_3) Iron oxide, from the ironstone and any slag in the aggregate,
- **be** = Belite (C_2SiO_4) *di*-Calcium Silicate, clinker component in binder and occasionally found in slags,
- **zo** = Zoisite ($Ca_2Al_3(SiO_4)(Si_2O_7)(O,OH)_2$) Calcium Aluminium Silicate, hydraulic component in lime, and also a component of some coal clinker and iron slag materials (possible pozzolan),
- **wo** = Wollastonite (CaSiO3) Calcium Silicate, high temperature mineral, component of clinker or slag,
- **hy** = Hydrocalumite ($Ca_2Al(OH)_6Cl_2H_2O$) Calcium Aluminium Chloride Hydroxide Hydrate, hydration product, also known as Friedel's Salt, from hydration of clinker and in some pozzolanic reactions,
- **hm** = Hydromagnesite ($Mg_5(CO_3)_4(OH)_2H_2O$) Magnesium Carbonate Hydroxide Hydrate, hydration product of magnesium carbonate, component of limestone, some ironstones and in slag,
- **gy** = Gypsum ($Ca(SO_4)2H_2O$) Calcium Sulphate Hydrate, Sulphate reaction product from a reaction between environmental sulphates, or from the coal ash, acid rain, etc., and lime from the binder,
- **ill** = Illite clay mineral from the alteration of mica and alkali feldspars, present as an aggregate or weathering component.

The results from the XRD analysis were further processed using Rietveld Refinement, in the MAUD computer program. This permitted quantification of the components present to help in assessing the form of binder present and/or the hydraulic or pozzolanic hydration reactions that may be present.

The following results were obtained from the Rietveld Refinement:

Component	**Proportion (% by Mass)**				
Sample:	**SR2706-S1A RESK1A**	**SR2706-S1B RESK1B**	**SR2706-S2A RESK2A**	**SR2706-S2B RESK2B**	**SR2706-S3 RESK3**
Calcite	44.6	21.5	54.8	55.4	57.9
Aragonite	34.6	21.7	21.1	13.4	25.1
Quartz	0.3	1.4	2.1	1.4	2.0
Hematite	4.0	11.0	8.3	5.8	6.0
Belite (C_2S)	1.1				
Zoisite	2.6	3.4			1.1
Wollastonite			0.8	1.3	
Hydrocalumite	4.1	2.4	5.7	3.6	2.3
Hydromagnesite	7.2	4.8	4.7	14.4	3.7
Gypsum	1.5	33.8	2.5	2.3	1.9
Illite				2.4	
Total	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**

From the XRD analysis, it is indicated that the mortar samples received are all representative of high Calcium lime, i.e. air lime (Non-Hydraulic lime) mortars, with the variations between those examined and analysed being a function of the composition of the components of the bank slag (waste material) used as the aggregate in each.

The high Aragonite content is due to the type of limestone incorporated with the aggregate and perhaps, to some extent, the partially burnt limestone fragments that may have been incorporated with the quicklime.

The presence of pozzolanic components and clinker, in the form of Belite, Wollastonite and Zoisite, are considered to be components of the waste material from the iron kilns, that have been used as aggregates in the mortars. These are all high temperature minerals/products and, on the basis of the condition of the quicklime inclusions observed, it is considered that it is unlikely that they originated from the quicklime used in the mortar production. As it would not be expected that the limestone kilns would have reached sufficient temperature for their formation, though the presence of "hot spots" is not uncommon and, therefore, this source cannot be totally discounted.

The Hematite will have originated from the slag waste and ironstone observed in the aggregate, with the Hydrocalumite and Hydromagnesite again having most likely formed from the hydration of the potentially reactive (pozzolanic) components in the slag when mixed, and slaked, with the quicklime. This would also suggest that the mortars had been mixed as Hot Lime Mortars (HMM), though not necessarily placed as such.

A high gypsum content was noted in all samples, with this particularly noticeable in sample SR2706-S1B (RESK1B). The source of the sulphates necessary for its formation, from a reaction between the lime in the binder and sulphate, being either environmental sulphates, or the presence of sulphate minerals in the slag or the pyrite in the limestone and shale. The microscopic examination of the samples may help to clarify this.

Mix Composition

The results of the composition analysis was determined by acid digestion, on all samples except sample SR2706-S1A (RESK1A), as there was insufficient material available in this sample. The results of the analysis carried out are presented below:

Sample Ref. No.	**SR2706-S1B (RESK1B)**		**SR 2706–S2 (RESK2)**		**SR2706-S3 (RESK3)**	
Mortar type (from XRD)			Non-Hydraulic Lime			
Binder/Aggregate Ratio	1.0 : 0.8		1.0 : 1.0		1.0 : 0.6	
Binder form:	Quicklime	Hydrate	Quicklime	Hydrate	Quicklime	Hydrate
Weight proportions calculated mix ratio by dry mass.						
Lime	1.0	1.0	1.0	1.0	1.0	1.0
Aggregate	1.5	1.1	1.8	1.3	1.0	0.8
Approximate volume Proportions calculated on the basis of a Non-Hydraulic lime						
Lime	1.0	1.0	1.0	1.0	1.0	1.0
Aggregate	0.4	0.4	0.5	0.5	0.3	0.3

Sample Reference	**SR2706– S1B (RESK1B) Masonry Mortar**		**SR2706– S2 (RESK2) Masonry Mortar**		**SR2706– S3 (RESK3) Masonry Mortar**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**	**Percentage Retained**	**Percentage Passing**	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100	0	100	0	100
4.00mm	25.8	74.2	3.3	96.7	10.0	90.0
2.00mm	9.6	64.6	16.6	80.1	9.9	80.1
1.00mm	12.0	52.6	19.5	60.6	9.7	70.4
0.500mm	8.5	44.1	13.0	47.6	8.2	62.2
0.250mm	13.9	30.2	15.2	32.4	13.3	48.9
0.125mm	10.8	19.4	10.4	22.0	14.9	34.0
0.063mm	8.3	11.1	6.7	15.3	9.8	24.2
Passing	11.1		15.3		24.2	

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

The residue from the acid digestions were recovered by vacuum filtration, washed and dried with their particle size distribution determined. The results obtained are presented in table No. 1 above. With the gradings displayed in the form of aggregate filled histograms in the appended Figures No. 1, 2, and 3, shown at the end of this report.

The natural aggregates in the mortar are dominated by opaque minerals, limestone fragments with trace proportions of quartz and indeterminate lithic fragments within the silt/clay fractions.

The opaque minerals appear to be mostly waste from the Iron processing, i.e. ironstone, coal, coal clinker, ash, brick fragments along with overburnt limestone fragments.

Microscopic Examination

To permit further clarification of the form in which the binder was used, and comparison of the fabric in all samples, a petrographic thin section was prepared from each for examination in the polarised light microscope.

To achieve this a slice was sawn from the largest intact piece in each sample of the mortar, with these dried and impregnated with a blue dyed resin in preparation for manufacturing thin sections.

Observations from the examination of the thin sections are presented below:

Sample SR2706-S1A (RESK1A): Kiln 1, Vault 4 Brickwork – Pinkish White mortar

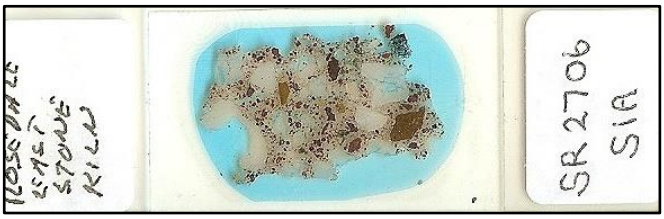

Plate No. 9:

Thin section prepared from mortar sample SR2706-S1A (RESK1A).

Aggregate

The aggregates in the mortar sample are dominated by coal, coal clinker and burnt limestone fragments, along with ironstone, and minor shale and brick fragment, with a trace proportion of very fine quartz grains. The latter possibly, components from the rock burnt in the processing of the lime or ironstone and/or from soiling contamination.

The coal fragments are a mixture of fresh particles, with no evidence of having been burnt, and partially burnt coal along with some coal clinker. Brick fragments are rare, sub-angular in shape with rounded margins and 0.2 to 0.7mm in size and all display weathering and are considered to be fragments from the kiln linings, included in the kiln waste materials.

The aggregates are angular to sub-angular and elongate in shape, with a high proportion displaying sharp margins. In addition, most show evidence of having been exposed to high temperatures and, locally, subsequent weathering. It is, therefore, concluded that they had been sourced from the waste material from the Iron processing kilns, that had been crushed, and possibly screened. Though a proportion of waste from the limestone kilns may also have been included.

The overburnt limestone observed is mostly from a bioclastic/Oolitic limestone and although burnt it had not been sufficiently calcined and the rock fabric was clearly visible. These particles had not been sufficiently burnt to function as binder.

It was also noted that some of the shale particles and flaky limestone particles observed contained an abundance of fine pyrite crystals, and these may be the source, at least in part, of the sulphate that had reacted with the lime to form the gypsum detected.

Binder

The binder is a non-hydraulic lime, with the abundance, of angular and sub-angular lime inclusions observed suggesting that the mortar had been mixed as a hot mixed mortar (HMM) and the quicklime had not been run to putty before use.

The paste is fully carbonated with the lime inclusions observed ranging from angular, sub-angular to sub-round, and occasionally irregular, in shape, with a low proportion of the inclusions also showing features consistent with the presence of partially slaked quicklime within the mix at the time of placing.

The inclusions range in size from 0.4mm to 3.7mm and it is inferred from the examination of these, and the encapsulating paste, that the quicklime was most likely slaked to a hydrate whilst mixed with the sand. However, in the absence of the features commonly associated with hot placed mortars, the mix may have been slaked to a dry condition (hydrate) before re-tempering and placed as a cold mortar mix.

Voids and microcracks

The voids observed are mostly irregular and, locally elongated in shape, and are placing artefacts. Localised angular voids appear to have formed from the dissolution of lime inclusions. The voids range in size and shape, from 0.2mm to 3.1mm, and many of these retain a patchy coating of fine calcite crystals with clusters of sulphate minerals. The latter mostly observed lining surface connected voids, indicative of water percolation through the voids.

Cracks are abundant and are random in occurrence and distribution. These are mostly typically of drying shrinkage features. The cracks are variable in length and typically <0.04mm in width. Cracks are commonly lined with secondary mineral deposits, including calcite and gypsum.

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

Sample Ref:	**SR2706-S1A (RESK1A)**	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	0.2	0.2
Limestone	12.2	12.2
Ironstone	10.9	10.9
Siltstone/shale	2.7	2.7
Brick	0.6	0.6
Opaque, coal/clinker/ash	12.0	12.0
Lime inclusions	-	23.0
Total Aggregate	**38.6**	**61.6**
Binder (Lime)	35.0	35.0
Clinker	0.6	0.6
Lime inclusions	23.0	-
Secondary products/Calcite and gypsum	2.8	2.8
Total Binder	**61.4**	**38.4**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**7.0**	**7.0**
	Total	**Effective**
Binder: Aggregate Ratio	**1.0 : 0.6**	**1.0 : 1.6**

Table No. 2: Result of modal analysis (300-point count) on thin section from RESK1A.

Photomicrographs:

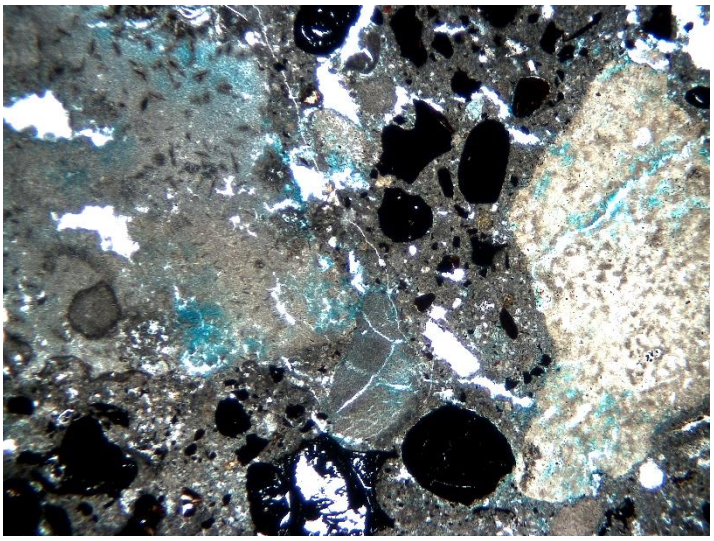

Plate No. 10:

A view in plane polarised light (ppl) showing a typical area of the mortar, with a partially hydrated inclusion seen in the left of centre and a limestone fragment on the right side. The paste is dense and fully carbonated, with no evidence of hydraulic clinker observed within this area. The aggregates are dominated by opaque particles (black in image), with fine ash grains distributed throughout the paste. Voids can be seen both within the lime inclusion and throughout the paste, along with fine shrinkage cracks. Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

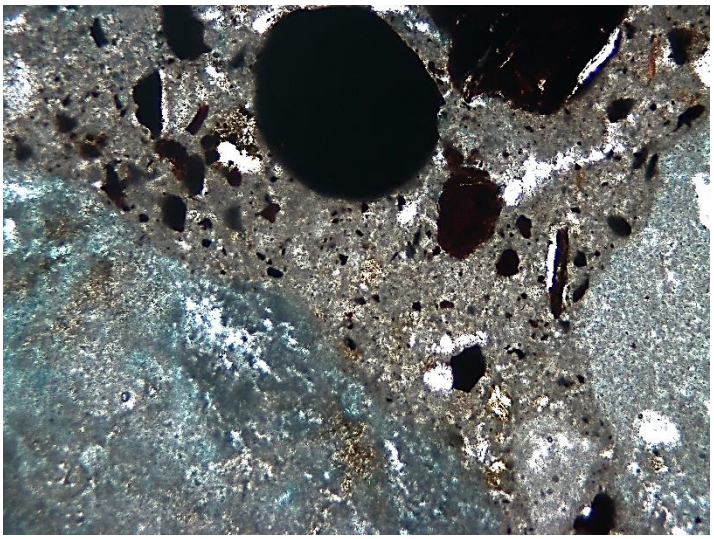

Plate No. 11:

A magnified view in ppl, of an area of paste where two inclusions can be seen, centre right, partially hydrated and lower left fully slaked and hydrated. Coal fragments can be seen in the upper part of the plate along with angular shards of coal clinker and slag materials distributed through the paste. Some of the fine grains show reaction rims and there are pseudo-morph Belite grains apparent in the centre of the plate, both inferring that the mortar has hydraulic/pozzolanic hydrates present. Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

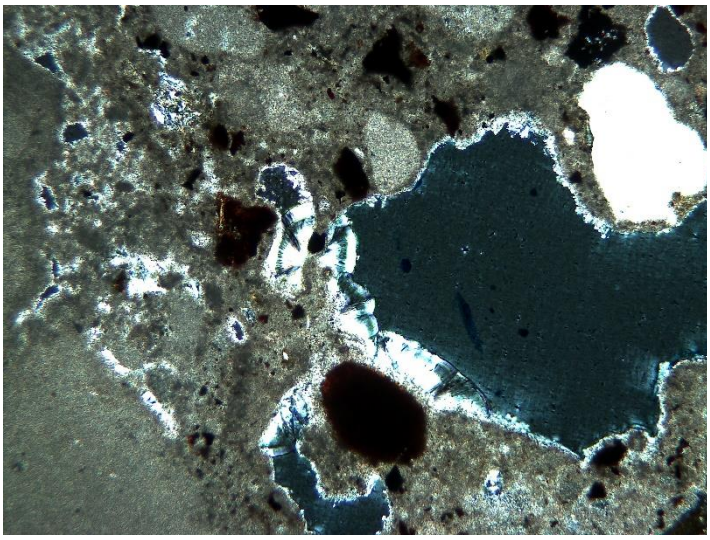

Plate No. 12:

A view in cross polarised light (xpl), of an area of dense fully carbonated paste. An irregular shaped void in the right of centre is rimmed with fine calcite with clusters of sulphate minerals in its lower left side (both white in image). The connecting channel ways, to the left are also lined with secondary minerals confirming water migration over time. Small lime inclusions can be seen in upper left with a larger inclusion in the lower left corner. The impregnating resin, porosity and voids all show dark in xpl. Field of view 1.2mm.

Sample SR2706-S1B (RESK1B): Kiln 4, Vault 13, Brickwork – White Mortar

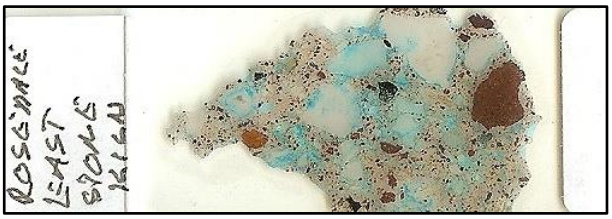

Plate No. 13:

This section was prepared from an intact piece of mortar from sample SR2706-S1B (RESK1B).

This sample is visually very similar to sample S1A (RESK1A), in that the aggregates and lime inclusions are of the same materials, with the following modal analysis carried out to confirm mix composition for comparison.

From the microscopic examination the main difference between this sample and sample S1A (RESK1A) was the abundance of pyrite minerals observed in the shale fragments and some flaky limestone fragments. This may be the cause of the higher proportion of sulphate minerals observed lining voids and the wider crack paths within this sample. Sulphates were also noted to locally permeate into the paste adjacent to the crack paths.

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

Sample Ref:	**SR2706-S1B (RESK1B)**	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	0.2	0.2
Limestone	9.5	9.5
Ironstone	10.5	10.5
Siltstone/shale	0.5	0.5
Brick	2.3	2.3
Opaque, coal/clinker/ash	13.4	13.4
Lime inclusions	-	23.1
Total Aggregate	**36.4**	**59.5**
Binder (Lime)	35.9	35.9
Clinker	0	-
Lime inclusions	23.1	-
Secondary products/Calcite and gypsum	4.6	4.6
Total Binder	**63.6**	**40.5**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**8.0**	**8.0**
	Total	**Effective**
Binder: Aggregate Ratio	**1.0 : 0.6**	**1.0 : 1.5**

Table No. 3: Result of modal analysis (300-point count) on thin section RESK1B

Photomicrographs:

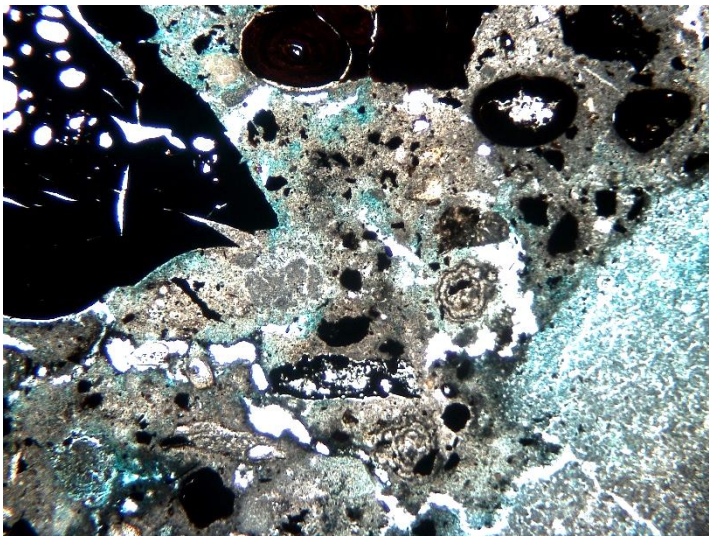

Plate No. 14:

A view in plane polarised light (ppl) of a typical area of the mortar. This shows the dense fabric with localised alteration of paste adjacent to opaque minerals. A lime inclusion fills the lower right corner with a partially burnt coal fragment in the upper left corner. Fine coal, ironstone and clinker grains are distributed throughout the paste as are small lime inclusions (putty) centre, and lower left. A number of the finer clinker particles display reaction rims, and these are taken to indicate that a pozzolanic reaction has occurred.

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

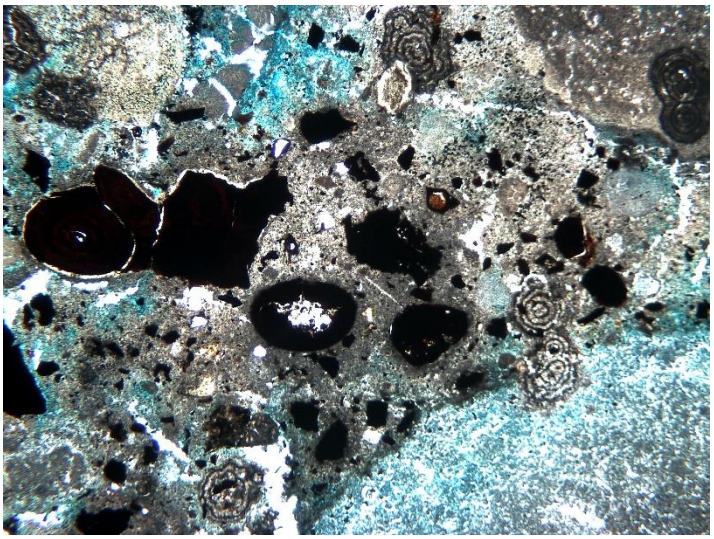

Plate No. 15:

A magnified view in ppl of an ash and slag rich area of fabric packed between two lime inclusions, one to the lower right the other upper left. A limestone fragment can be seen in the upper right corner. The paste is fully carbonated and contains an abundance of coal, ash, clinker and fine ironstone fragments. Note the pattern of Liesegang rings, right of centre at margin with an inclusion and in the lower centre, these are indicative of a change in differential morphology in response to cyclic reprecipitation of binder components. Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

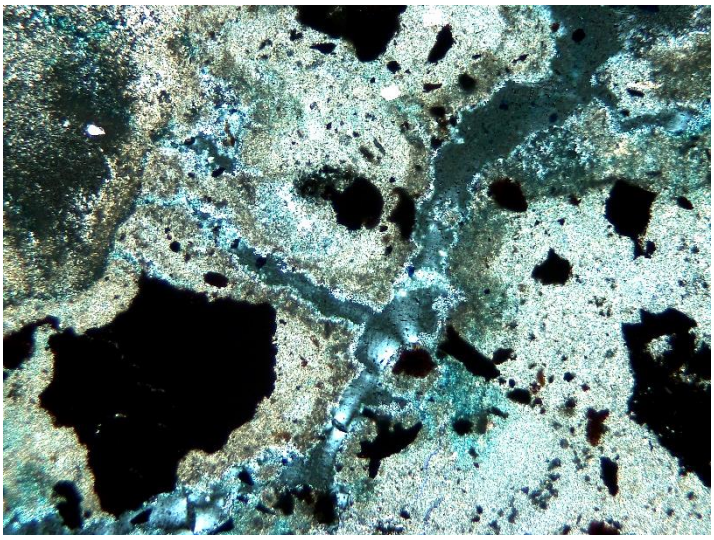

Plate No. 16:

A view in Cross Polarised Light (xpl), of an area of dense carbonated paste transected by a wide crack which has acted as a water percolation channel. Note the discolouration of the paste either side of the crack indicative of depletion with the redeposition of calcite and coarse sulphate minerals within the crack path. The paste on the left, lower quarter also shows sulphate minerals diffused through the paste around the large void in this area. The upper left shows a partially slaked and partially hydrated lime inclusion, with ash clusters in the upper right corner. Porosity, the blue impregnating resin and opaque minerals all appear black in xpl. Field of view 1.2mm.

Sample SR2706-S2 (RESK2): Kiln 4, Mortar from Rear Retaining Wall – Pink Mortar

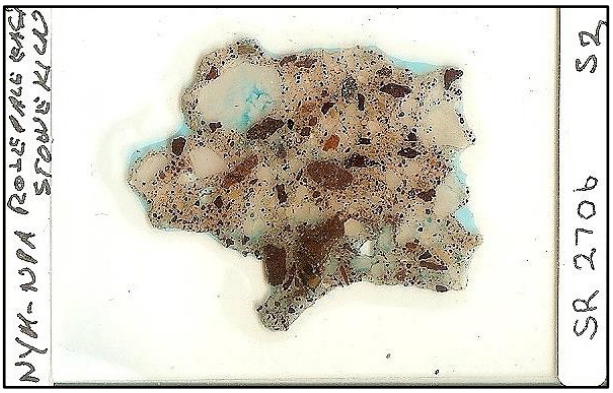

Plate No. 17:

This thin section was prepared from an intact piece of mortar from sample SR2705-S2 (RESK2).

This sample had the appearance of a well compacted mortar, with a dense fabric, having a relatively uniform appearance throughout its thickness. However as with samples SR2706S1A & B the components present appear to be the same, with only variations in the proportions of each being observed.

The results of a point count (modal) analysis carried out to confirm mix composition are presented in the following table:

Sample Ref:	**SR2706-S2 (RESK2)**	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	0.3	0.3
Limestone	11.2	11.2
Ironstone	7.2	7.2
Siltstone/shale	3.3	3.3
Brick	1.1	1.1
Opaque, coal/clinker/ash	10.1	10.1
Lime inclusions	-	18.7
Total Aggregate	**33.2**	**51.9**
Binder (Lime)	46.2	46.2
Clinker	0	-
Lime inclusions	18.7	-
Secondary products/Calcite and gypsum	1.9	1.9
Total Binder	**66.8**	**48.1**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**5.0**	**5.0**
	Total	**Effective**
Binder: Aggregate Ratio	**1.0 : 0.50**	**1.0 : 1.1**

Table No. 4: Result of modal analysis (600-point count) on thin section from RESK2

Photomicrographs:

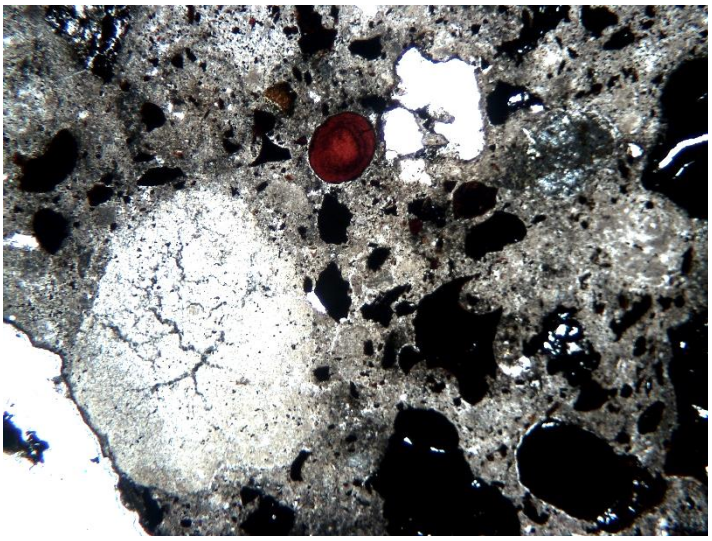

Plate No. 18:

A view in plane polarised light (ppl) showing a well compacted area of the mortar, where the paste is fully carbonated and is relatively dense. A fully slaked lime inclusion is apparent in the lower left quarter. The inclusion displays shrinkage cracking but has not diffused into the surrounding paste. Aggregates in view are mostly opaque minerals and include coal, ironstone fragments, with coal clinker and ash. A mapwork of very fine shrinkage cracks are apparent, around the perimeter of aggregates and distributed throughout the paste. Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

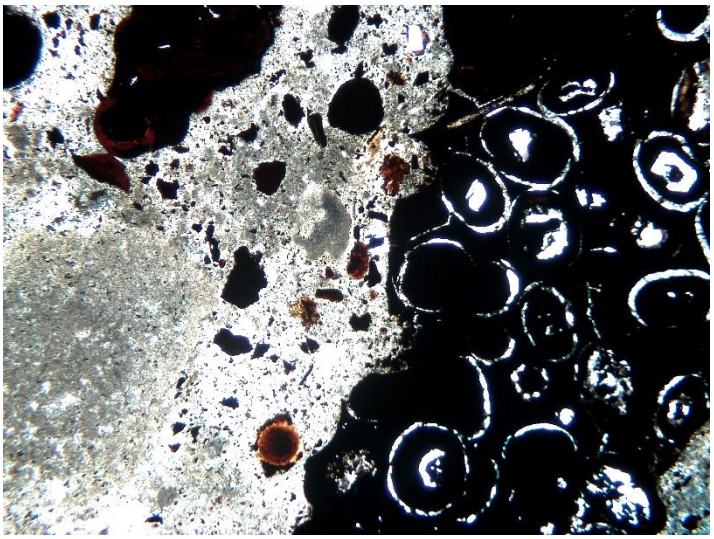

Plate No. 19:

A view in plain polarised light (ppl) of a dense area of paste, left side of image, in which a fully slaked lime inclusion can be seen, this has an angular outline, and it is fully encapsulated in a fully carbonated paste. This contains an abundance of opaque particles. The right side of the image is filled by a partially burnt ironstone particle. The paste in the upper centre also contains an abundance of fine ash particles, none of which appear to have reacted with the paste at this location, i.e. no pozzolanic activity. Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

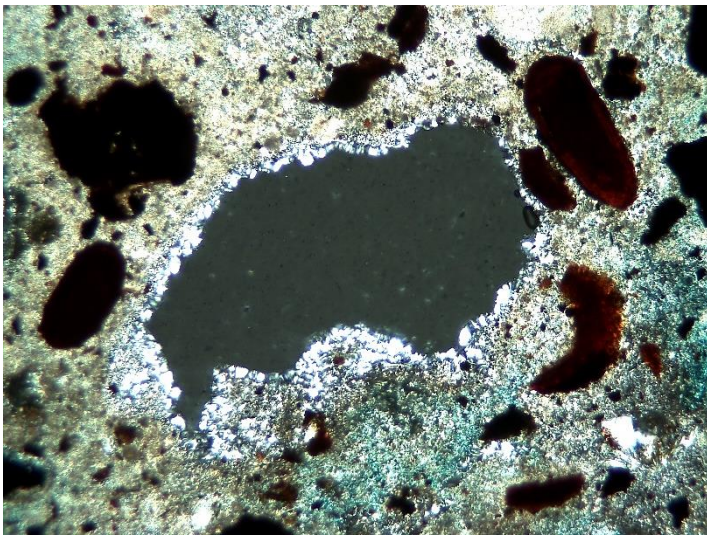

Plate No. 20:

A view in xpl, of a void rimmed with a heavy growth of secondary minerals dominated by calcite with minor sulphate minerals. The surrounding paste is fully carbonated and contains small coal and clinker fragments along with ash clusters, the latter mostly in the lower centre and right. A patchy discolouration can be seen adjacent to a number of the opaque minerals and this may be indicative of pozzolanic reactions having occurred.
Porosity, the blue impregnating resin and opaque minerals all appear black in xpl. Field of view 1.2mm.

Sample SR2706-S3 (RESK3): Kiln 4, Mortar from Front Wall/Facing – White Mortar

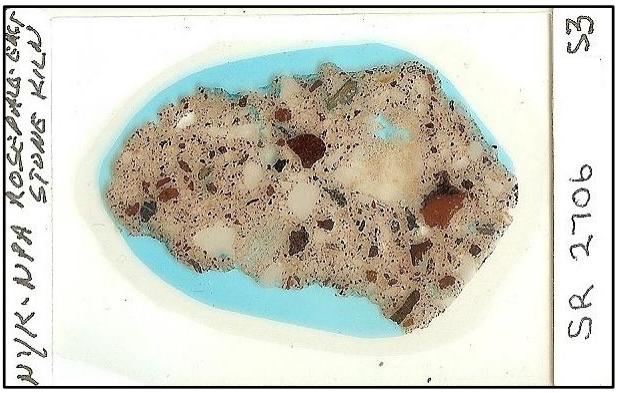

Plate No. 21:

This thin section was prepared from an intact piece of mortar from sample SR2706-S3 (RESK3).

This sample is again visually similar to the sections made from samples S1A & B and S2 (RESK1A & B and RESK2).

This sample is very similar to the previous samples and it is indicated, visually, to have been made from the same materials.

Aggregate

The aggregates are very similar to those in the earlier samples examined from this batch of samples in that it is composed mostly of coal, coal clinker, ash, slag and ironstone with a trace proportion of brick fragments and quartz grains. This sample contained several fragments of overburnt ironstone measuring up to 6.4mm in size and burnt limestone up to 6.2mm in size.

Again, the presence of the slag and ash will have resulted in the mix having a high pozzolan content, with its reaction resulting in the strength of the hand specimen. With the source of this considered to be the waste or spoil heaps adjacent to the Iron kilns.

The aggregates are angular to sub-angular and locally irregular in shape. With the maximum size of being 7.2mm but mostly finer than 1mm down to <0.02 mm (ranging from medium silt to very coarse sand and medium gravel size) in the section examined. The presence of an abundance of very fine grains observed is consistent with aggregates containing a significant proportion of ash and crushed, or ground, coal ash clinker, slag and brick dust.

Binder

The paste in this sample is again dense with the presence of an abundance of inclusions suggesting that the mix had been mixed as a Hot Mixed Mortar (HMM). This sample also contains a high proportion of partially burnt and partially slaked quicklime particles, a number of which have not diffused into the surrounding paste and are, therefore, acting as aggregates in the mortar and not contributing as a binder.

However, the sample also has the appearance of a binder rich mix and the paste is locally very dense, with depletion of binder, due to leaching, being restricted to the margins of cracks and water percolation pathways, and very locally within the cores of larger lime inclusions. The paste is fully carbonated.

On the basis of the fabric condition, the abundance of lime inclusions and the localised evidence of densification around inclusion margins, it is inferred that this mortar had been both mixed and either placed as a hot mixed mortar (HMM), or had undergone delayed slaking of incompletely slaked quicklime, after placing.

The presence of slag, ash and burnt ironstone fragments in the mortar would account for the hardness and strength of the sample with the evidence of the reactivity of the fine aggregate (ash/slag) grains inferring that pozzolanic reactions had occurred.

Voids and microcracks

Voids are present, and range up to 2.8mm in size, they also vary from irregular voids typical of placing artefacts to smaller angular and sub-rounded voids formed from the depletion of quicklime lime, and putty, inclusions. All voids retain, at least in part, a lining of redeposited calcite.

Cracks are abundant in this sample and are mostly wider than 0.02mm and locally extend up to 0.2mm in width. A high proportion of the cracks link lime inclusions and skirt around coarse aggregate particles, with these having the appearance of plastic and early drying shrinkage features.

All of the wider cracks are lined with secondary minerals, including both coarse calcite crystals and fine needle shaped sulphate minerals. These channel ways, therefore, appear to have functioned as water percolation channel ways, with water having accessed the mortar through them over an extended period of time.

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

Sample Ref:	**SR2706-S3 (RESK3)**	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	0.5	0.5
Limestone	17.0	17.0
Ironstone	12.0	12.0
Siltstone/shale	1.2	1.2
Brick	1.0	1.0
Opaque, coal/clinker/ash	14.2	14.2
Lime inclusions		11.1
Total Aggregate	**45.9**	**57.0**
Binder (Lime)	40.0	40.0
Clinker	0.6	0.6
Lime inclusions	11.1	-
Secondary products/Calcite and gypsum	2.4	2.4
Total Binder	**54.1**	**43.0**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**4.3**	**4.3**
	Total	**Effective**
Binder: Aggregate Ratio	**1.0 : 0.85**	**1.0 : 1.3**

Table No. 5: Result of modal analysis (600-point count) on thin section from RESK3

Photomicrographs:

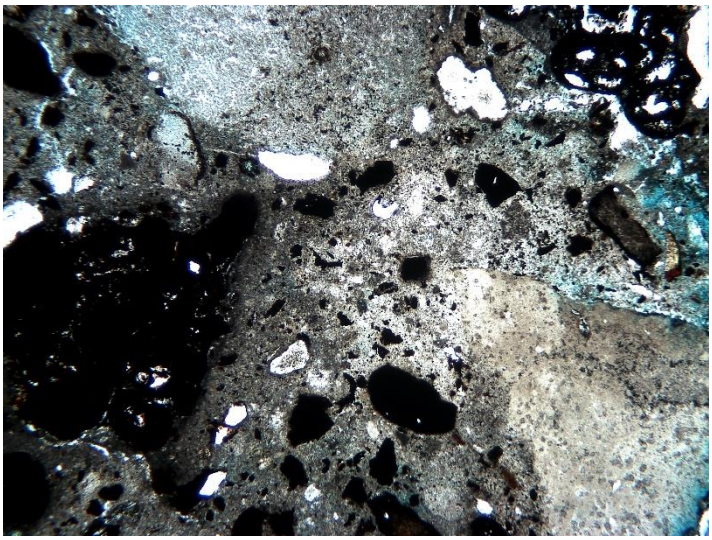

Plate No. 22:

A view in plane polarised light (ppl) of a typical area of the mortar. This shows a partially calcined and partially slaked, but not diffused, lime inclusion in the right side of the plate, this has performed as an aggregate in the mortar. A fully calcined but partially slaked, but locally diffused lime inclusion can be seen in the upper left. Aggregates in view include coal ash clinker, lower left, with fine coal and ironstone fragments, lower centre, upper left and right. Ash clusters can be seen in the centre and upper right of centre. The paste is dense and is fully carbonated. Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

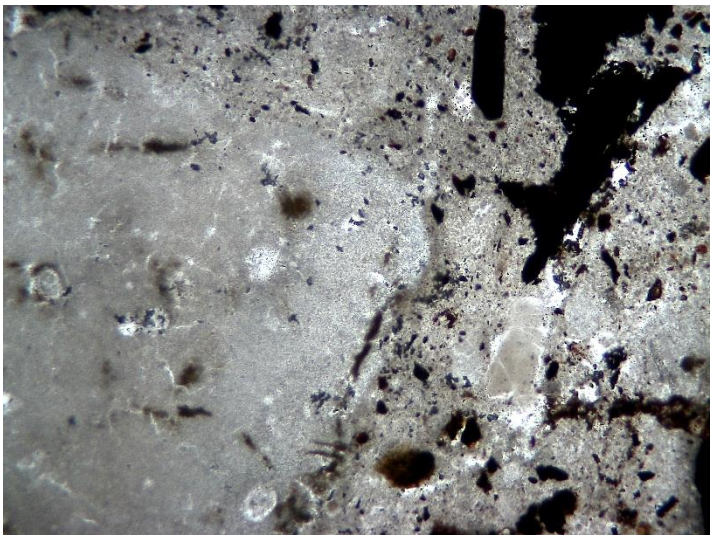

Plate No. 23:

A magnified view in ppl of fully slaked lime inclusion which fills the left part of the plate. Although fully calcined it is not fully hydrated. This contains some fine impurities. This inclusion displays a sharp outer margin and acts as an aggregate and does not diffuse into the paste. The paste in the right and upper centre is dense and well compacted, it is fully carbonated and contains small patches of fully hydrated lime (putty). Aggregates in view are mostly coal clinker and ash, with only minor evidence of any reactions occurring. Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

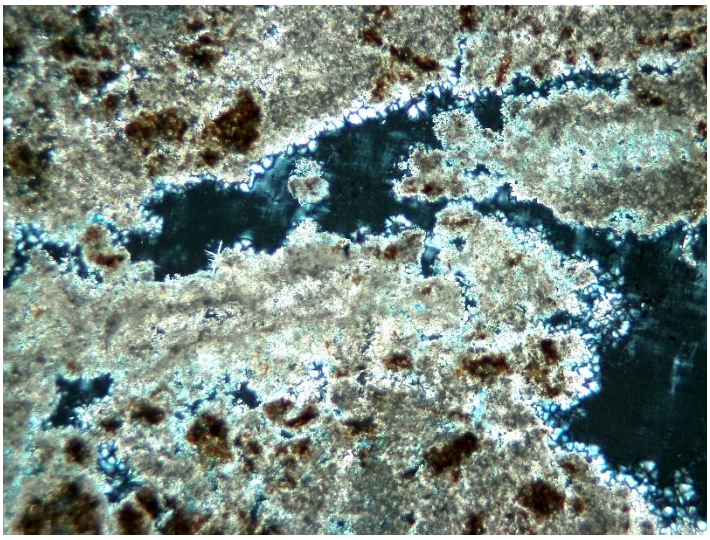

Plate No. 24:

A view in Cross Polarised Light (xpl), of an area of the mortar containing wide cracks that have acted as water percolating channel ways. The binder is locally depleted in the adjacent paste with coatings of calcite and gypsum crystals providing a white fringe to the channel way margins and connecting voids. The paste contains minor clinker and ash grains, some of which show reaction rim, indicating that a pozzolanic reaction had occurred.
Porosity, the blue impregnating resin and opaque minerals all appear black in xpl. Field of view 1.2mm.

Summary

From the examination and analysis of the four mortar samples received from the Rosedale East Stone Kilns, it is indicated that the mortars sampled were all from the same, or very similar mortar mix prepared from a high calcium lime and mixed with waste material from the Iron processing kilns as aggregate.

The mortar in these samples all appears to have been prepared from the quicklime being mixed with the aggregate (bank slag) and the quicklime slaked, as in a Hot mixed Mortar (HMM). Although, in most samples there was no evidence to confirm that the mortar was placed as a hot mix mortar, and quicklime was most likely slaked to dry condition, with the dried mortar mix stored and remixed prior to use in the form of a cold mixed mortar. With the presence of an abundance of quicklime inclusions and partially burnt limestone fragments in the mortar also suggesting that it may not have been screened, through a fine mesh screen, prior to remixing.

However, locally there is evidence of some late slaking of the lime within the mortars, and it is considered that this is due to the late slaking of individual particles, post placing, rather than suggest that the mortar was placed as a hot mortar.

The lime used in all four samples is a high calcium lime produced by calcining a local limestone, with this having a fabric consistent with a micritic limestone (micro spar), with minor bioclastic limestone also present.

The fuel in the firing of the lime kiln may have been coal, with the possibility that a low proportion of the unburnt/partially burnt fuel was carried over into the mortars, possibly as a contaminant, with the quicklime. Nonetheless, it is indicated that the majority of the coal, both fresh and partially burnt, along with coal clinker, ash and ironstone, and partially burnt limestone had originated from the waste from the Iron processing kilns used as the aggregates in these mortars.

The mortars all show the impact of a pozzolanic reaction between reactive components in the aggregate, in the coal clinker, ash and iron slag, though this is patchy in occurrence, it has imparted a degree of strength to the mortars, albeit they all continue to display a moderate to high microporosity, which along with shrinkage cracks and primary and secondary voids will permit the mortars to be porous and vapour permeable.

All of the mortars show the impact of sulphate reactions, with the formation of gypsum. The source of the sulphate required for this reaction is in part the pyrite observed within finely laminated shale and limestone fragments observed in the aggregate, and possibly from external environmental sources. Locally the impact of sulphate reducing bacteria was also observed within the paste in samples SR2706-S1B and S2 (RESK1B and RESK2). The presence of the high sulphates in the mortar is not necessarily a problem, unless it is proposed to use Portland type cements in any future repair or restoration works, where their presence could be disruptive to the new mortars.

A summary of the mortar mixes determined is reproduced below:

Sample Ref. No.		**RESK1A**	**RESK1B**	**RESK2**	**RESK3**
Binder form:		Quicklime	Quicklime	Quicklime	Quicklime
Approximate volume proportions calculated on the basis of a Non-Hydraulic lime					
Mix composition by Acid Digestion					
Lime : Aggregate Ratio			1.0 : 0.4	1.0 : 0.5	1.0 : 0.3
Mix composition by Modal Analysis					
Lime : Aggregate	Total	1.0 : 0.6	1.0 : 0.6	1.0 : 0.5	1.0 : 0.8
	Effective	1.0 : 1.6	1.0 : 1.5	1.0 : 1.1	1.0 : 1.3

The effective binder content determined from the modal analysis is calculated on the basis that the inclusions are acting as aggregate rather than binder, and this is probably a truer measure of the binder content of the mix, relating to its performance as a mortar. Whereas, the total lime content reflects the mix composition at the time the mortar was made and placed, including the inclusions as part of the added lime binder, and reflects the mix proportioning at the time of mixing.

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 samples of mortar received in CMC's laboratory on the 12th March 2019 from Structural & Civil Consultants Ltd., which were identified as mortars from the Rosedale East Stone Kilns, in the North Yorkshire National Park.

W A Revie
For CMC Ltd.

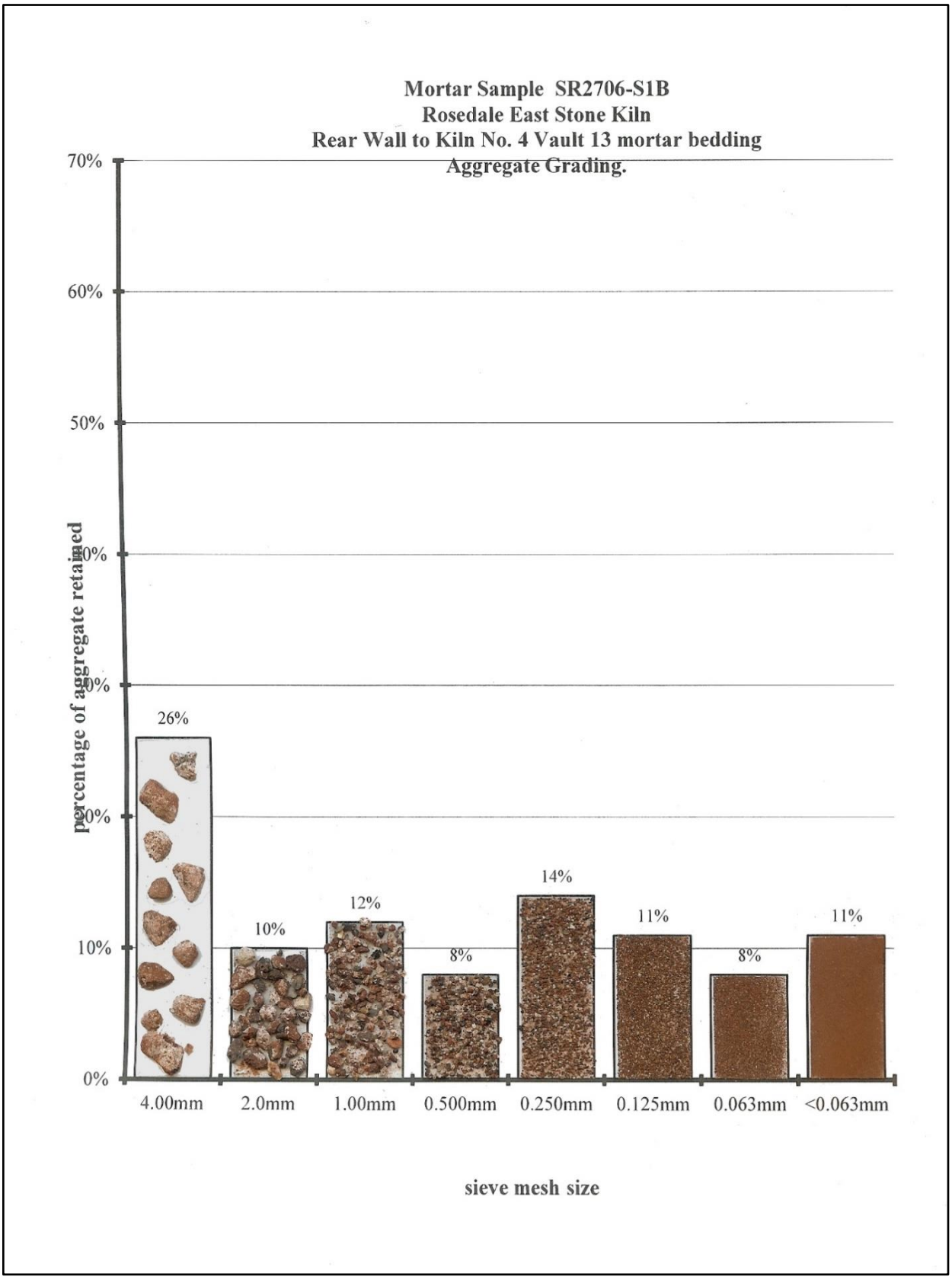

Figure No. 1: Aggregate Grading on Aggregate recovered from sample RESK1B.

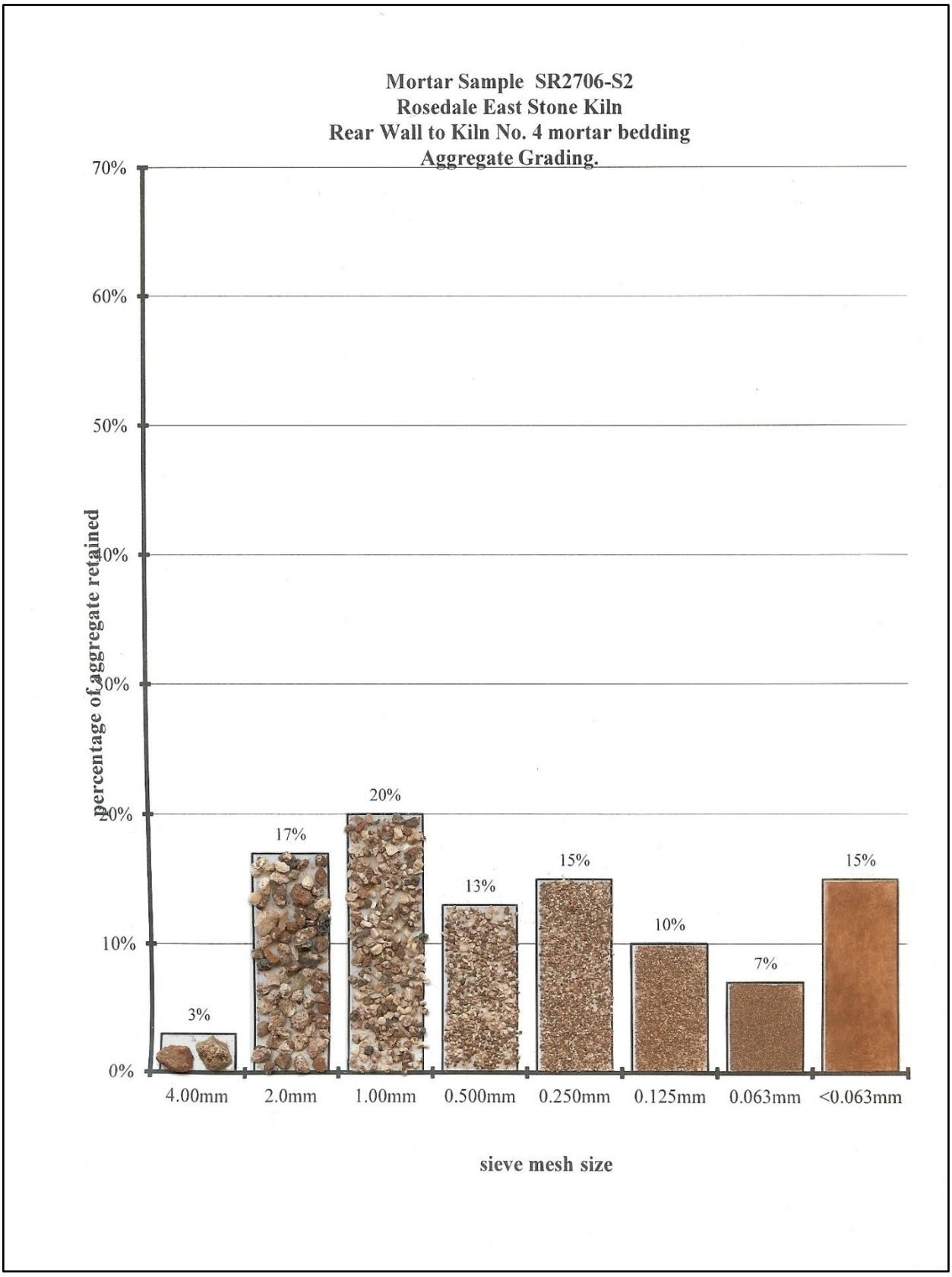

Figure No. 2 – Aggregate Grading on Aggregate recovered from sample RESK2.

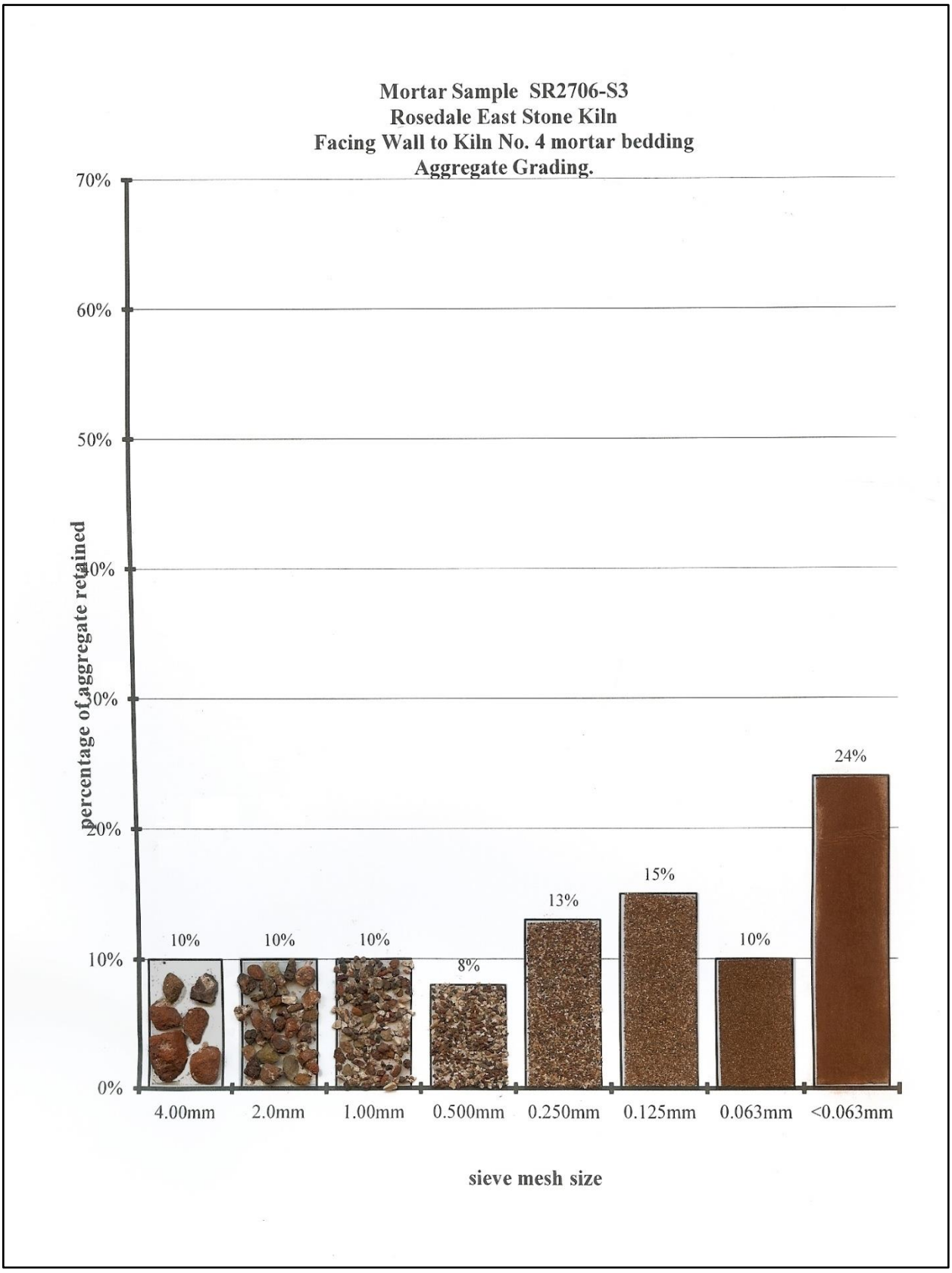

Figure No. 3 – Aggregate Grading on Aggregate recovered from sample RESK3.

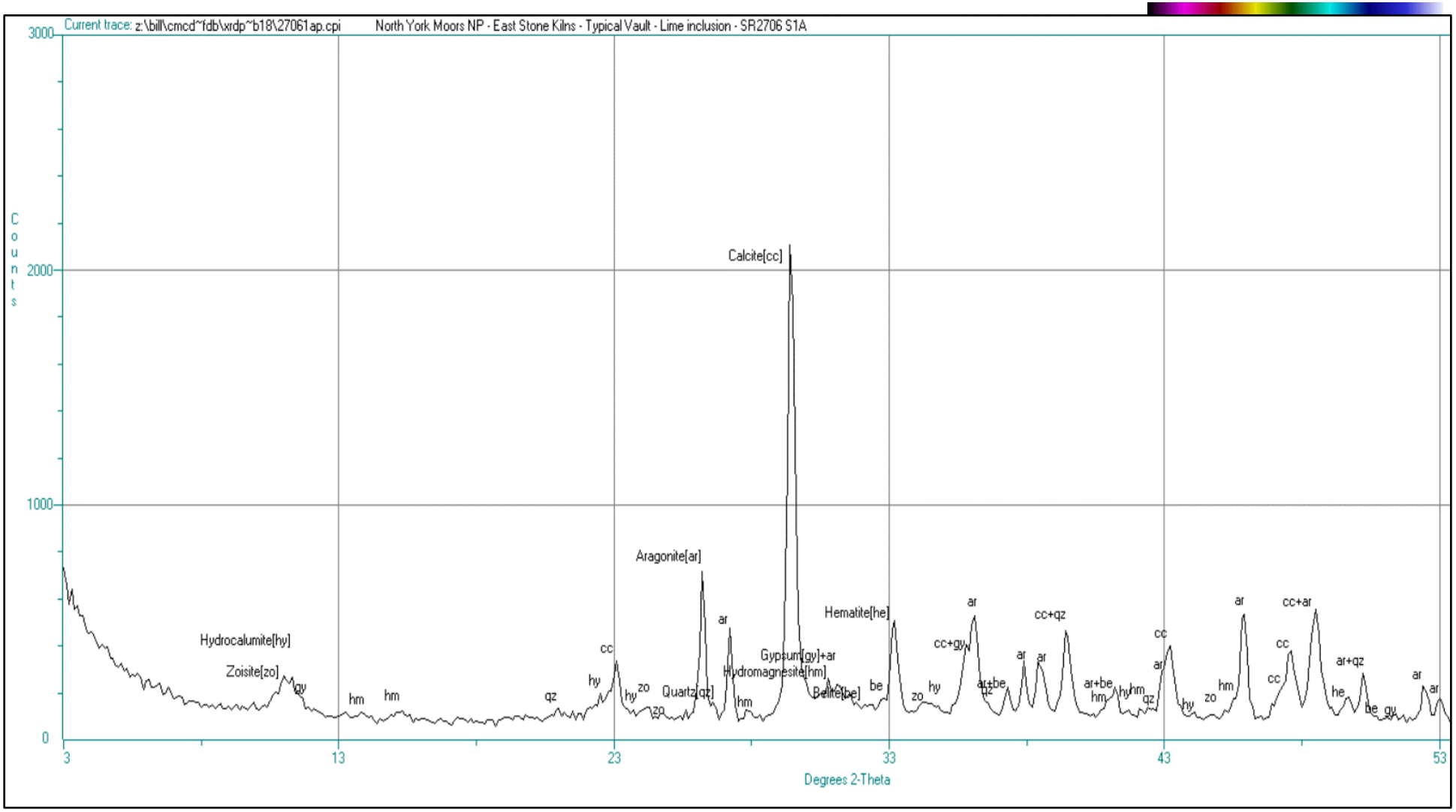

Figure No. 4: SR2706-S1A (RESK1A) Kiln 1, Vault 4, Brickwork, Pinkish White mortar.

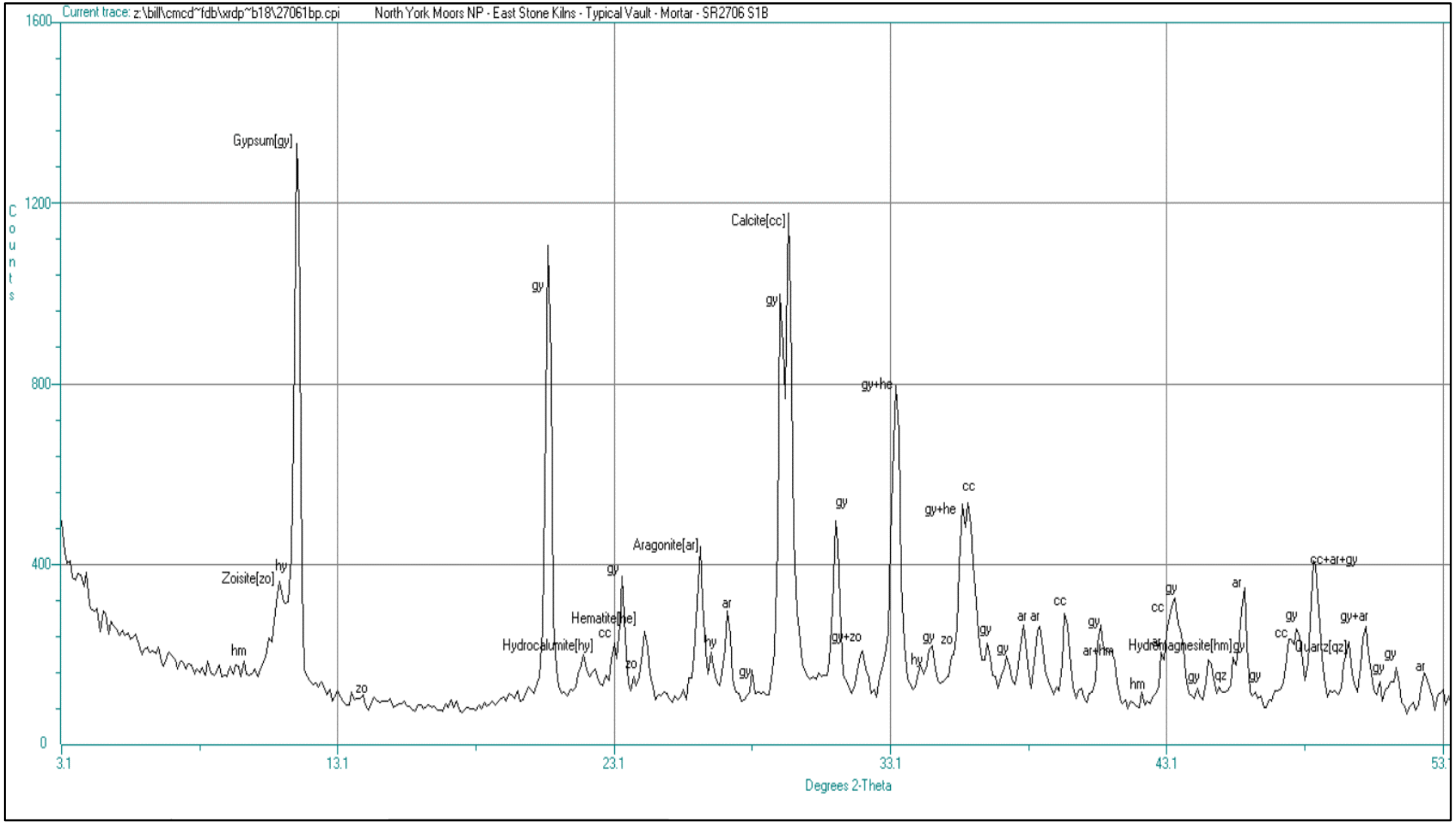

Figure No. 5: SR2706-S1B (RESK1B) Kiln 4, Vault 13, Brickwork, White mortar,

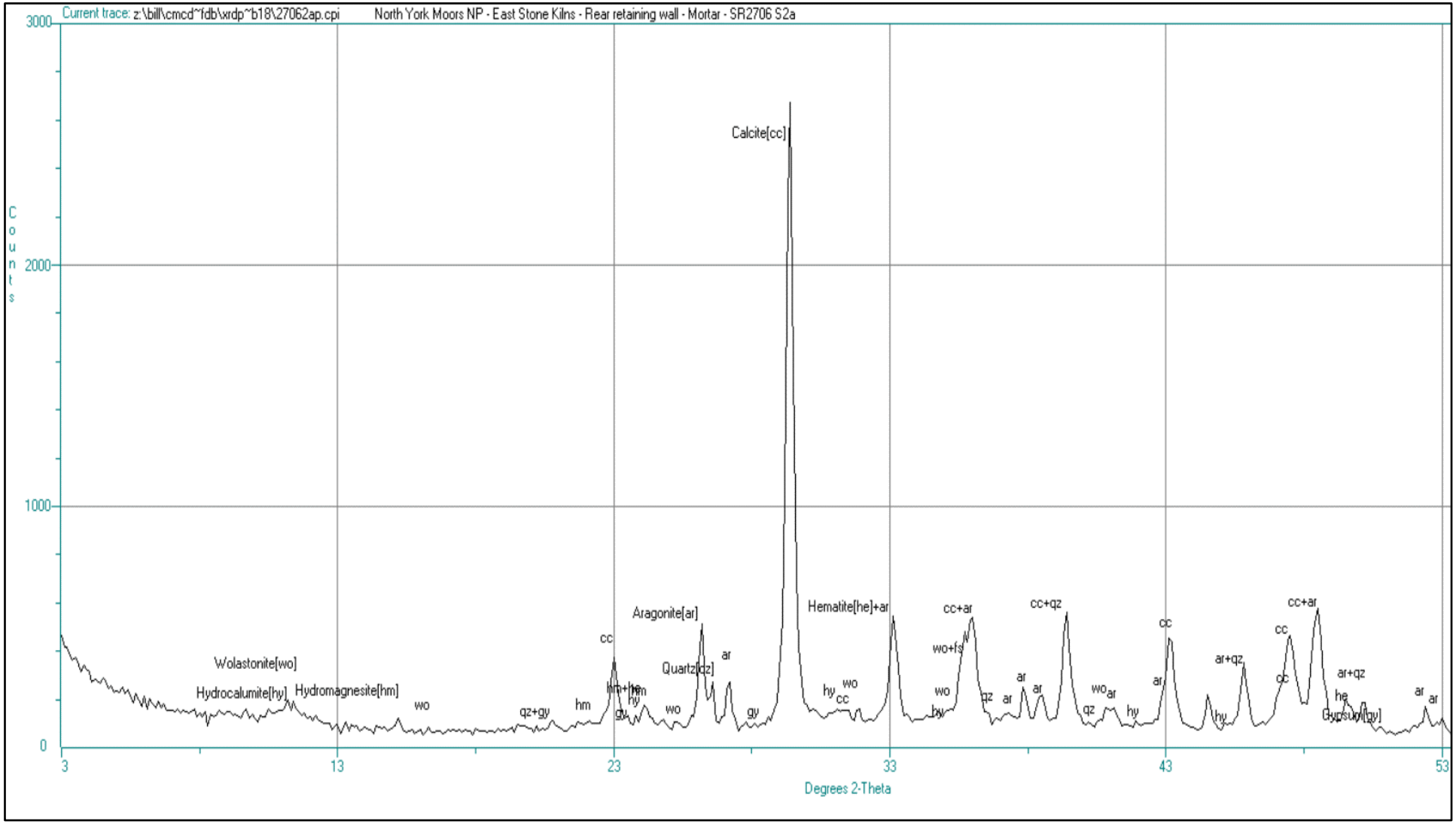

Figure No. 6: SR2706-S2A (RESK2A) Kiln 4, Rear Retaining Wall, Friable Pink mortar.

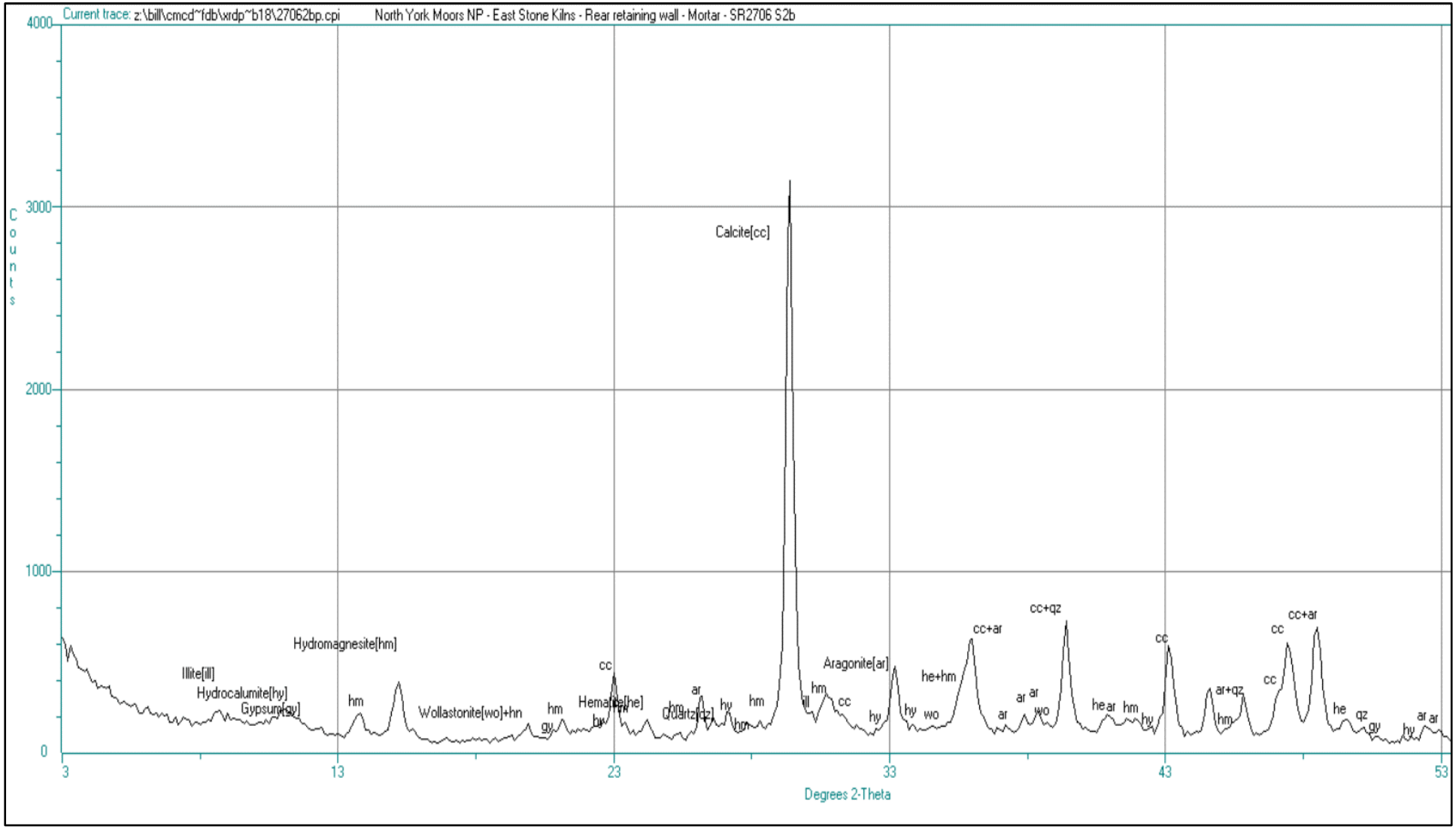

Figure No. 7: SR2706-S2B (RESK2B) Kiln 4, Rear Retaining Wall, Hard Pink mortar.

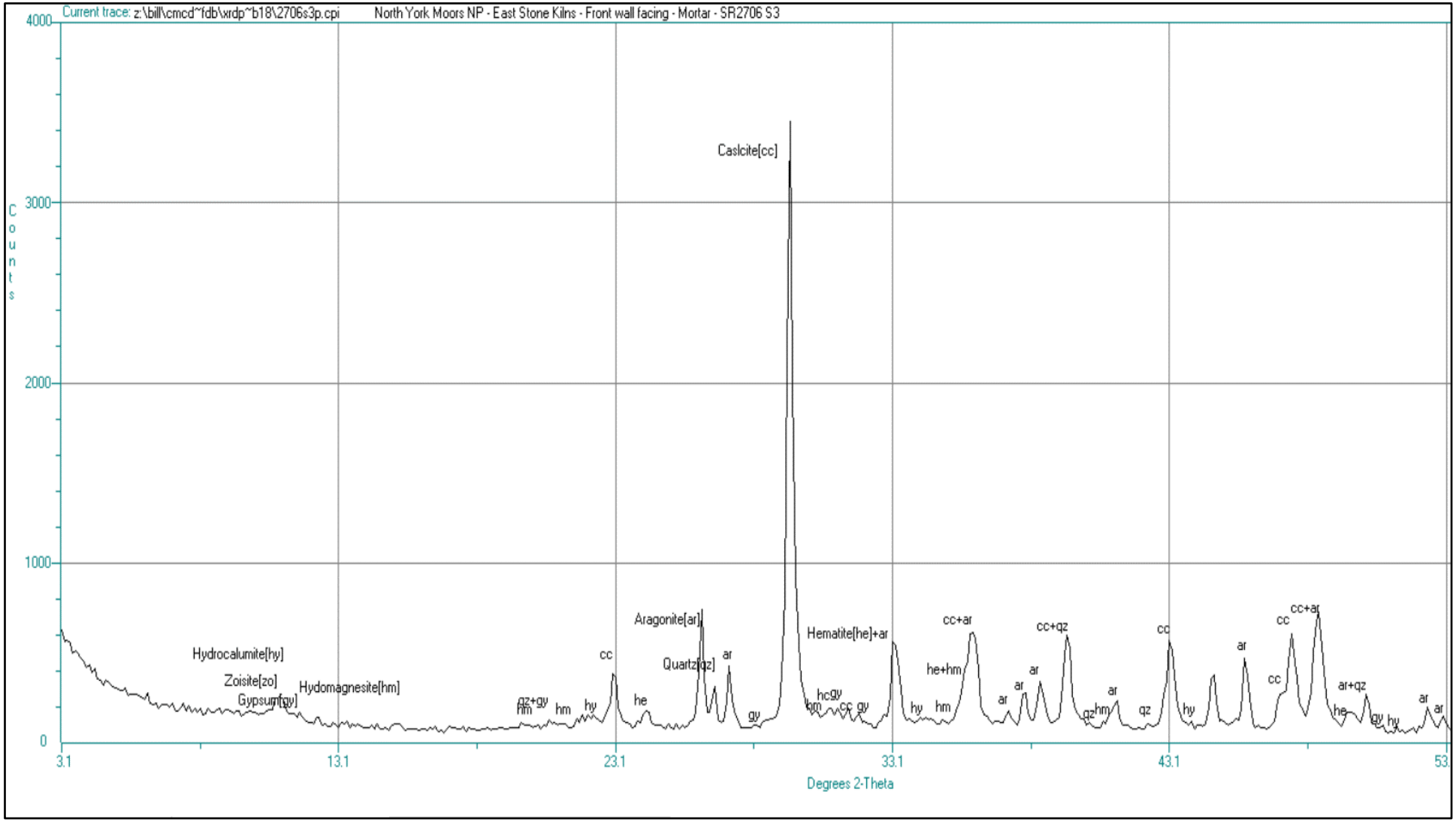

Figure No. 8: SR2706-S3 (RESK3) Kiln 4, Front Wall/Facing, White bedding mortar.