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Our Ref: M/1991/19/C5  
Your Ref.: T2019

5<sup>th</sup> 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 Iron Kilns
Sample Description	:	REIK1 – Mortar from Fire Brick Lining REIK2 – Mortar from Pier 1 Stonework
Date Received	:	12 <sup>th</sup> March 2019
CMC Sample Ref	:	SR 2707-S1 = REIK1 – Mortar from Fire Brick Lining SR 2707-S2 = REIK2 – Mortar from Pier 1 Stonework
Date Analysed	:	10 <sup>th</sup> April to 30 <sup>th</sup> 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

Two mortar samples were received from the Rosedale East Iron Kilns, with the samples 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 received in CMC's Stirling Laboratory on the 12<sup>th</sup> March 2019, as part of a batch of samples from this project and were to be submitted to analysis to determine the composition of the mortar in each sample. In addition, the grading of the aggregates was to be determined and comment offered on the condition of the mortars as received, and, if possible, on the type of binder 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 SR2707 allocated.

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

CMC Sample Ref.	Client Ref	Location Sampled
SR2707 – S1	REIK1	Mortar from Fire Brick Lining in the Iron Kiln
SR2707 – S2	REIK2	Mortar from Pier Stonework, in the Iron Kiln.

CMC



## 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 which the samples were exposed to a series of *ad hoc* droplet tests employing a range of reagents and indicator solutions.

Following the initial examination, a binder rich sub-sample was obtained from each sample for X-ray Diffraction (XRD) analysis. This was to permit identification of the binder type used in the production of each mortar. This analysis technique was used 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, a representative sub-sample from SR2707-S1 (RSIK1) was prepared for mix composition by acid digestion, following the methods of the Scottish Lime Centre Trust (SLCT). Sample SR2707-S2 (REIK2) was too small to permit composition analysis by acid digestion and this was achieved by modal analysis. The insoluble residue remaining after the acid digestion of sample REIK1 was recovered by vacuum filtration, washed to remove excess acid, dried and sieved through a nest of British Standard sieves. This permitted the particle size distribution of the aggregate to be determined.

In addition to the above a petrographic thin section was prepared from each sample. This would permit clarification of the form in which the binder was used, along with providing confirmation of the mix composition by modal analysis, in the event that there were any acid soluble aggregate components present in the mortars.

## 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
SR2707-S1	REIK1	245.7	114.8 x 60.2 x 11.2	2.5YR 5/2 "Weak Red"
SR2707-S2	REIK2	30.7	54.0 x 25.3 x 10.5	10R 6/2 "Pale Red"

### Sample SR2707-S1 (REIK1): Mortar from Fire Brick Lining – Iron Kilns

This sample consisted of eight fragments of a light red coloured mortar with all fragments well compacted and moderately hard. 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. It was noted that the freshly fractured surfaces were not to be friable, and aggregates well bonded.

On examination the mortar was noted to contain an abundance of dark minerals in the aggregate, with a few small lime inclusions also observed, these were typically <1.0mm in size and irregular in shape.

The outer surfaces of the samples were heavily soiled with localised patches of fines from the surface of the kiln brickwork adhering to the mortar.

On testing a freshly fractured surface with phenolphthalein indicator the mortar was found to be fully carbonated.

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From the water droplet tests, where droplets were applied to both outer surfaces and freshly fractured surfaces, it was indicated that the mortar had a low connected porosity, with the droplets supported with only minor shallow absorption noted, after two minutes exposure.

Spot tests with dilute Hydrochloric acid produced a strong effervescent reaction, as would be expected on a carbonated lime mortar, but this was accompanied by a strong odour of hydrogen sulphide (H<sub>2</sub>S), which was emitted, indicating that the sample perhaps contained pyrite or sulphur rich coal clinker.



**Plates No. 1 & 2:** The left plate shows the intact pieces in the mortar sample, as-received. With the right plate showing a close-up image of a freshly fractured surface through the thickness of the sample. Note compact nature of the mortar and the brick fragments adhering to the upper surface of the mortar, to which it was very well bonded.

The aggregates appear to be dominated by dark minerals with coal and brick fragments also apparent. With the largest aggregate particle observed being in the region of 5.5mm in size, however most of the aggregate particles are less than 1.0mm.

On testing the aggregates coarser than 0.063mm, recovered from the sample prepared for XRD, it was noted that a high proportion of the fines were attracted to a magnet, indicating a high ironstone or slag content.

**Sample SR2707-S2 (REIK2): Mortar from Pier Stonework**

This sample contained several small fragments 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, but once broken the fractured surfaces were noted to be finger friable and aggregates picked from the surface with ease.

The mortar in all fragments were noted to contain an abundance of lime inclusions, which were distributed throughout. The inclusions are mostly angular to sub-round in shape and measure up to 6.1mm in size. On probing the inclusions, the majority were found to be soft and easily picked out, although there were a number that were harder and resisted disruption under a point pick, and this may infer that there were over or under burnt limestone fragments present.

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





**Plates No. 3 & 4:** The left plate shows the sample as-received, which was mostly of small mortar fragments along with a quantity of fines. The right plate shows a freshly fractured surface through the thickness of the largest fragment, in which an abundance of lime inclusions, and possibly limestone fragments were apparent. Small dark coal and brick fragments were observed randomly distributed throughout.

A fragment of sandstone adhered to one of the outer surfaces, on some fragments, indicating that a good bond had formed between the mortar and the masonry of the Pier to which it was applied.

The aggregates are sub-angular to sub-round and irregular to elongate in shape and are dominated by coal and ironstone, with clinker and ash along with a proportion of small limestone fragments also noted.

### Results of XRD Analysis for Binder Type

To help clarify the composition of the binder in the mortar, a representative sub-sample was obtained from each sample. These were crushed in an impact mortar 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 powdered sub-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  $3^\circ$  to  $60^\circ$   $2\theta$  in steps of  $0.1^\circ$   $2\theta$  at a rate of  $1^\circ$   $2\theta$ /minute using  $\text{CuK}\alpha$  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  $0.1^\circ$ .

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

**Figure No. 2:** SR2707-S1 (REIK1) Mortar from Fire brick Lining – Iron Kiln,

**Figure No. 3:** SR2707-S2 (REIK2) Mortar from Pier Stonework – Iron Kiln.

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

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- cc** = Calcite ( $\text{CaCO}_3$ ) Calcium Carbonate, carbonated lime from lime binder and any limestone aggregate present in the mortar,
- ar** = Aragonite ( $\text{CaCO}_3$ ) another crystalline form of Calcium Carbonate, from limestone, commonly associated with shell and found in some forms of redeposited leached lime binder,
- va** = Vaterite ( $\text{CaCO}_3$ ) a further crystalline form of Calcium Carbonate, from limestone and commonly associated with carbonated redeposited, leached lime.
- qz** = Quartz ( $\text{SiO}_2$ ) Silicon Oxide, a component of the aggregate in some of the mortars,
- he** = Hematite ( $\text{Fe}_2\text{O}_3$ ) Iron oxide, from the ironstone and any slag in the aggregate,
- be** = Belite ( $\text{C}_2\text{SiO}_4$ ) *di*-Calcium Silicate, clinker component in binder and occasionally found in slags,
- zo** = Zoisite ( $\text{Ca}_2\text{Al}_3(\text{SiO}_4)(\text{Si}_2\text{O}_7)(\text{O},\text{OH})_2$ ) Calcium Aluminium Silicate, hydraulic component in lime, and also a component of some coal clinker and iron slag materials (possible pozzolan),
- fr** = Friedel's Salt ( $\text{Ca}_4\text{Al}_2\text{O}_6\text{Cl}_2\cdot 10\text{H}_2\text{O}$ ) Calcium Aluminium Oxide Chloride Hydrate, hydration product, from the hydration of clinker and in some pozzolanic reactions,
- ett** = Ettringite ( $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}$ ) Calcium Aluminium Sulphate Hydroxide Hydrate, hydration product in Portland Cements and some Hydraulic limes, also sulphate reaction product,
- gy** = Gypsum ( $\text{Ca}(\text{SO}_4)\cdot 2\text{H}_2\text{O}$ ) 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,
- di** = Dickite, clay mineral of the Kaolinite group of minerals, from the alteration of Alkali Feldspar, present as an aggregate component, or a weathering product.

The results from the XRD analysis were processed using Rietveld Refinement, in the MAUD computer program, which permitted quantification of the individual crystalline components.

The results obtained are shown below:

Component Sample:	Proportion (% by Mass)	
	SR2707-S1 REIK1	SR2707-S2 REIK2
Calcite	47.1	14.1
Aragonite	3.4	7.8
Vaterite	21.4	
Quartz	1.6	3.2
Hematite	15.8	12.6
Belite ( $\text{C}_2\text{S}$ )	3.5	
Friedel's Salt	2.3	
Zoisite		7.3
Ettringite	4.2	
Gypsum		55.0
Dickite	<u>0.7</u>	<u>      </u>
<b>Total</b>	<b>100.0</b>	<b>100.0</b>

From the XRD analysis, it is indicated that the mortar in the two samples differ with respect to their aggregate composition, albeit both are indicated to be lime mortars, made with an air lime. The strength in sample SR2707-S1 (REIK1) is considered to be due to the presence of an abundance of pozzolanic components in the aggregate, with their reaction indicated by the abundance of hydration products present.

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Examination and Analysis of Mortar samples.



Sample SR2707-S2 (REIK2) although again made with a high Calcium lime, the binder in this sample appears to have undergone severe sulphate attack, with most of the lime now converted to gypsum, in the sample analysed. The source of the sulphate required for this reaction may have been from within the aggregate, but also in percolating waters or from flue gasses arising from the burning process.

The presence of pozzolanic hydration products in the form of Friedel’s Salt, in both samples, and clinker, in the form of Belite, in sample REIK1, are considered to be due to the presence of the coal clinker, ironstone and slag from Iron processing being used as the aggregate, rather than suggest a hydraulic lime in the binder.

The high Hematite content, in both samples, indicating a high proportion of ironstone in the aggregate, or perhaps heavy contamination with iron dust from the iron burning process, or both, with, perhaps, a small proportion of the hematite from the brick fragments in the aggregates.

**Mix Composition**

The results of the composition analysis was determined by acid digestion, on sample SR2707—S1 (REIK1) only, as there was insufficient material in sample SR2707-S2 (REIK2), to permit mix composition by this method. The results of the analysis carried out are presented below:

<b>Sample Ref. No.</b>	<b>SR2707-S1 (REIK1)</b>	
Mortar type (from XRD)	Non-Hydraulic Lime	
Binder/Aggregate Ratio	1.0 : 1.6	
Binder form:	Quicklime	Hydrate
Weight proportions calculated mix ratio by dry mass.		
Lime	1.0	1.0
Aggregate	2.8	2.0
Approximate volume Proportions calculated on the basis of a Non-Hydraulic lime		
Lime	1.0	1.0
Aggregate	0.8	0.8

Sample Reference	SR2707– S1 (REIK1) Masonry Mortar	
	Percentage Retained	Percentage Passing
8.00mm	0	100
4.00mm	16.1	83.9
2.00mm	17.9	66.0
1.00mm	1.3	64.7
0.500mm	6.9	57.8
0.250mm	15.4	42.7
0.125mm	16.6	25.8
0.063mm	11.4	14.4
Passing	14.4	

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

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The residue from the acid digestions were recovered its particle size distribution determined, with the result of the grading analysis presented in table No. 1 above. With the grading also presented in the form of an aggregate filled histogram in the appended Figure No. 1.

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

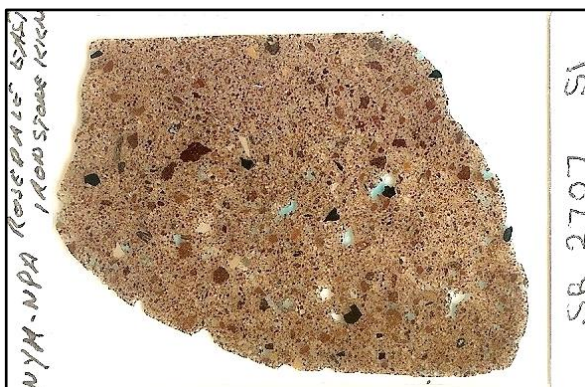
The opaque minerals appear to be mostly waste materials from the Iron processing, and are dominated by ironstone with coal, coal clinker, ash, and brick fragments along with minor overburnt limestone fragments.

**Microscopic Examination**

To permit further clarification of the form in which the binder was used, and permit comparison of the fabric in both 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 mortar, with these dried and impregnated with a blue dyed resin in preparation for the manufacture of thin sections.

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

**Sample SR2707-S1 (REIK1): Mortar from Fire Brick Lining – Iron Kiln**



**Plate No. 5:**

Thin section prepared from mortar sample SR2707-S1 (REIK1).

**Aggregate**

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

The coal fragments are a mixture of fresh unburnt particles, and partially burnt coal along with coal clinker. The coal fragments range in size from 3.2mm down to 0.07mm. Brick fragments are rare, these have rounded margins and evidence of weathering, typically <0.4mm in size. Ironstone fragments are again a mixture of fresh stone fragments and partially burnt fragments, with a proportion showing the impact of weathering, these range in size from 0.02mm to 6.4mm.

The brick fragments are present in allow proportion and it is not considered that the brick was added as a pozzolan, but rather as a component of the waste material used as aggregate.

Ash is present as fine fragments diffused through the paste and as small clusters of poorly bound grains.



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A low proportion of limestone fragments were observed, with these angular to sub-angular in shape and all show no evidence of having been burnt, they measure up to 2.0mm in size.

The aggregates are generally angular to sub-angular, locally rounded to elongate in shape, with a high proportion displaying sharp margins and these have the appearance of having been crushed. It is, therefore, likely that they had been sourced from the waste material from the Iron processing kilns, that had been crushed, and possibly screened.

***Binder***

The binder has the appearance of a non-hydraulic lime, with a proportion of sub-round to irregular lime inclusions observed. The lime inclusions are all well slaked, with most of the sub-rounded inclusions being fully hydrated, whereas a proportion of the denser fragments were only partially slaked and some of these retained a granular texture. This would suggest that the mortar had been mixed with a binder in the form of a hydrate. Locally the hydrated had formed concentrations which had 'balled' typical of those that form when a dry hydrate is mixed with damp sand.

The paste is fully carbonated throughout the sample, with evidence of localised redeposition of calcite and sulphate minerals (ettringite) infilling voids, some of which were formed by the post-placement dissolution of lime inclusions. This would infer moisture percolation through the sample over time.

The inclusions range in size from <0.02mm to 0.9mm and it is inferred from the examination of these, and the encapsulating paste, which displayed a patchy microporosity, indicating that the mortar was placed as a relatively workable mix.

Hydraulic components including Belite particles were observed, randomly distributed throughout the paste, but their occurrence was patchy, with most observed as pseudo-morph grains. However, there was no clinker apparent within the lime inclusions, which may, therefore, infer that the clinker had originated from another source, other than the lime. However, in the absence of other common hydraulic clinker components, and in the presence of pozzolanic materials (coal clinker, ash and slag from iron processing), a high proportion of which displays reaction rims and alteration, it is considered that the hydraulic/pozzolanic property of the mortar is a result of using the waste material from the iron processing operations as the aggregate.

***Voids and microcracks***

The voids observed are mostly irregular to sub-round, locally elongated in shape, and are a mixture of placing artefacts and as dissolution voids from the depletion of lime inclusions. The voids range in size and shape, with rounded voids ranging from 0.01mm to 1.1mm, whereas the irregular and elongated voids extend up to 3.4mm in size. All retain a thick fringe of coarse calcite crystals with clusters of sulphate minerals. In addition, very localised clusters of entrained air voids, spherical voids up to 0.3mm, were also observed, most of which now contain secondary minerals, both indicating that the mortar was initially mixed as a wet mix and was well worked.

Cracks are rare and are random in occurrence and distribution. These are mostly typically of drying shrinkage features. The cracks are variable in length and typically 0.01 to 0.05mm in width. Cracks are commonly lined with secondary mineral deposits, dominated by ettringite.

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



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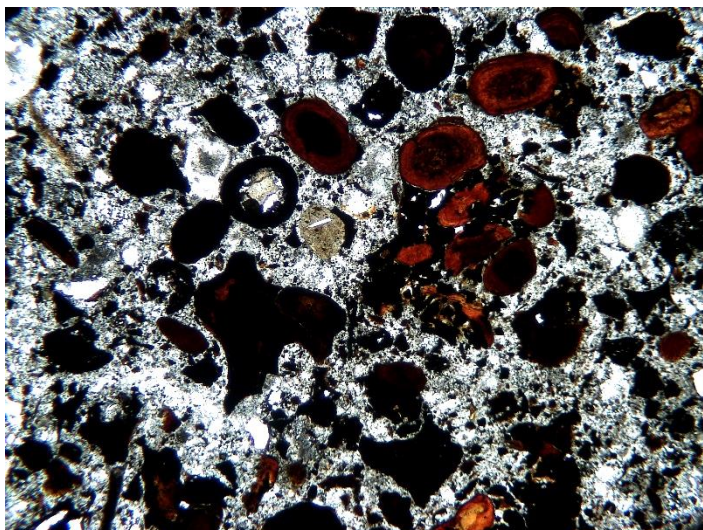
Examination and Analysis of Mortar samples.



Sample Ref:	SR2707-S1 (REIK1)	
Constituents	%	
Aggregate	Inclusions as binder	Inclusions as Aggregate
Quartz	0.7	0.7
Limestone	3.7	3.7
Ironstone	15.4	15.4
Siltstone/shale	0.3	0.3
Brick	0.7	0.7
Opaque, coal/clinker/ash	27.9	27.9
Lime inclusions	-	12.6
<b>Total Aggregate</b>	<b>48.7</b>	<b>61.3</b>
Binder (Lime)	36.6	36.6
Clinker	2.1	-
Lime inclusions	10.5	-
Secondary products/Calcite and sulphate minerals	2.1	2.1
<b>Total Binder</b>	<b>51.3</b>	<b>38.7</b>
<b>Total Constituents</b>	<b>100.0</b>	<b>100.0</b>
<b>Cracks/Voids</b>	<b>8.2</b>	<b>8.2</b>
<b>Binder: Aggregate Ratio</b>	<b>Total</b>	<b>Effective</b>
	<b>1.0 : 0.95</b>	<b>1.0 : 1.6</b>

**Table No. 2:** Result of modal analysis (600-point count) on thin section from REIK1.

**Photomicrographs:**

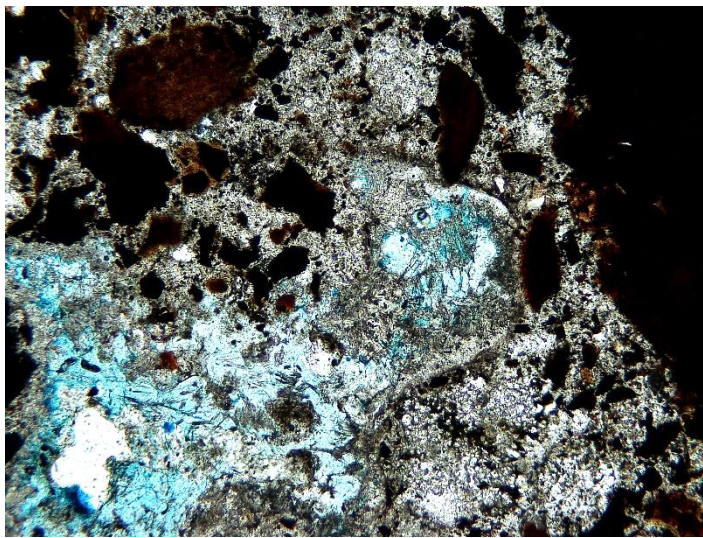


**Plate No. 6:**

A view in plane polarised light (ppl) of a typical area of the mortar, which shows the aggregates bound in a dense and fully carbonated paste. There are no lime inclusions observed in this area. The aggregates are dominated by opaque particles (black in image), with fine ash grains distributed throughout the paste. The coarser grains are dominated by coal fragments, coal clinker and ironstone (the latter brownish orange/black). Localised patches of microporosity are apparent, along with very fine angular quartz grains (white in image). Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

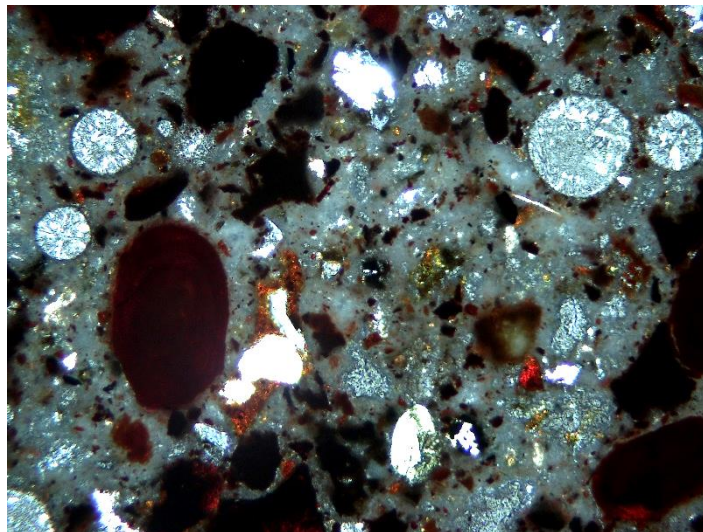
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Examination and Analysis of Mortar samples.



**Plate No. 7:**

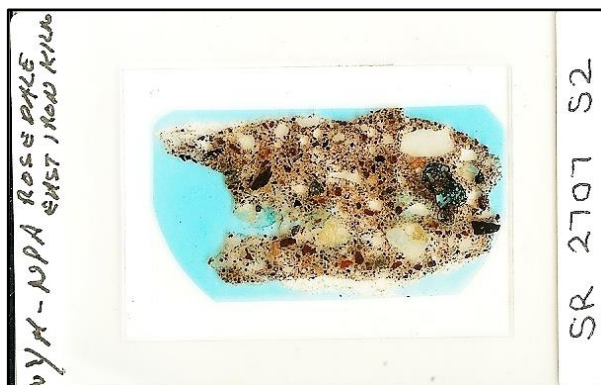
A magnified view in ppl, of an area of paste where the paste is partially depleted and both the small voids and the affected paste contain an abundance of secondary sulphate minerals. Some of the fine opaque grains show reaction rims and a pseudo-morph Belite grain is apparent in the upper centre of the plate, both inferring that the mortar has hydraulic and pozzolanic hydrates present. The right side of the plate is filled with a large ironstone fragment, with small particles also seen in the left side. Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.



**Plate No. 8:**

A view in cross polarised light (xpl), of an area of dense fully carbonated paste in which a number of entrained air voids (spherical) can be seen. These are all filled with sulphate minerals. Small ironstone fragments can be seen along with fine Belite grains, mostly upper right and right of centre. The alteration of fine coal clinker fragments, and slag particles, is highlighted by the loss of detail in the altered particles, with discolouration/alteration of the surrounding paste. The impregnating resin, porosity and voids all show dark in xpl. Field of view 1.2mm.

**Sample SR2707-S2 (REIK2): Mortar from Pier Stonework – Iron Kiln**



**Plate No. 9:**

This section was prepared from an intact piece of mortar from sample SR2707-S2 (REIK2).

The mortar in this sample is different to that in sample S1A (RESK1A), in that it is more binder rich, and has the appearance of an HMM, although it contains similar aggregate components, but in less quantity.



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Examination and Analysis of Mortar samples.



***Aggregate***

The aggregates in the mortar sample are again dominated by opaque minerals, with minor limestone with a low proportion of weathered brick fragments and very fine quartz grains

The limestone aggregates consist of both bioclastic and micritic forms i.e. algal/colloidal limestones. No Dolomitic limestone was observed in this sample. Some of the limestone fragments are partially burnt and may have been included with the quicklime binder, rather than the aggregate.

The opaque minerals are dominated by coal and coal clinker with a proportion of ironstone. The particles range from fresh, to weathered, fragments, with a proportion of both the coal clinker and the ironstone fragments displaying alteration. This would infer that they had originated from the waste material from the iron processing operations ongoing in this area of the moors.

Some of the coal fragments are partially burnt and locally these were observed bonded to partially burnt limestone, and, therefore, these particles are considered to be components from the lime kiln and were included as contaminants along with the quicklime.

Aggregates range in size from <0.1mm to 5.4mm, with irregular coal clinker particles measuring up to 5.4mm, angular coal fragments up to 3.2mm, ironstone up to 1.8mm and limestone 2.4mm. Most particles display sharp margins, and these may have been sourced from the crushed waste from the iron processing kilns, with, perhaps, minor components from lime burning kilns.

***Binder***

The binder has the appearance of a lime mortar, with a high abundance of lime inclusions observed within the section. Although there was evidence of potentially hydraulic components in the paste, its concentration and appearance would suggest that, in this sample, its occurrence is associated with the aggregate. As all of the lime inclusions, and the paste, have the appearance of having been formed lime a high calcium lime (non-hydraulic lime).

A quantity of the incompletely slaked inclusions retain a faint imprint of the original rock fabric, with both a bioclastic and micritic texture common. The lime inclusions observed are mostly angular to sub-angular, along with a minor proportion of sub-rounded inclusions. A number of the angular inclusions show features consistent with the limestone being either over or under burnt.

Significant areas of the paste display the effect of leaching, with the paste also displaying alteration, by the action of sulphates, perhaps introduced in percolating waters responsible for the leaching apparent. Patches of the paste are now converted to gypsum.

Although most of the inclusions observed in the section were completely slaked at the time of placing, there are indications that post-placing slaking had also occurred, though to a limited extent. The inclusions range in size from 0.5mm to 7.4mm and it is inferred from the examination of these, and the encapsulating fabric, that the quicklime was added to the sand in the form of a kibbled lime, or crushed lump lime, rather than powdered lime. With the quicklime slaked mixed with the sand, in the form of a Hot Mixed Mortar (HMM).

The presence of clusters of small entrapped air voids, centred on localised lime inclusions, with apparent compaction of the encapsulating paste, would also infer that a proportion of the mortar had been placed whilst it was still hot, and the lime was still undergoing slaking, therefore, it is indicated that the mortar was also placed as a hot lime mortar.

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Examination and Analysis of Mortar samples.



***Voids and microcracks***

Voids are rare and mostly irregular in shape with these being placing artefacts. Locally, sub-angular in shaped voids, typical of those arising from the depletion of lime from inclusions are also observed.

The voids range in size and shape, from 0.02mm to 1.2mm, with these typically irregular in shape, and formed in response to entrapped air. Most voids retain a fine crystalline coating of Calcite along with sulphate minerals. A patchy occurrence of localised clusters of entrained air bubbles were also noted. With these concentrated around some of the lime inclusions, the bubbles are mostly spherical to sub-rounded in shape and typically 0.02mm to 0.2mm in size.

Cracks are rare and occur as random features and locally adjacent to lime inclusions and at the interface with the adhering sandstone masonry. They are variable in length but range from <0.01mm to 0.05mm in width, and are typical of early drying, and plastic, shrinkage features. The crack paths are lined with fine calcite crystals with clusters of radiating needle-like crystals, typical of sulphate minerals, also present. Their presence would infer that water percolation through the crack paths had occurred over time.

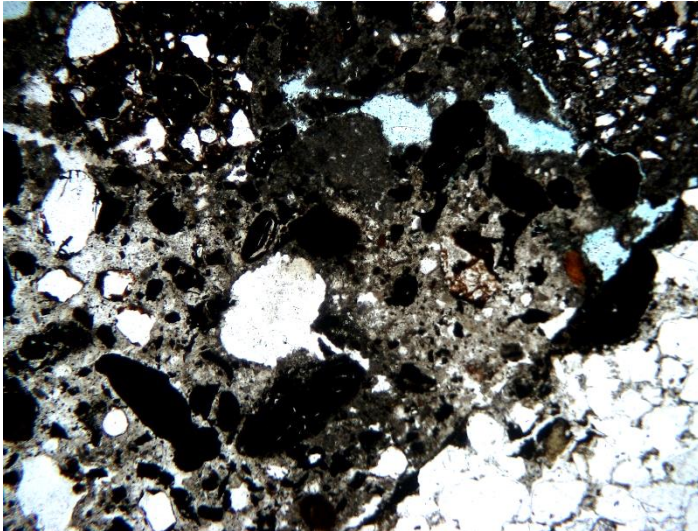
The results of a point count (modal) analysis is presented in the following table:

<b>Sample Ref:</b>	<b>SR2706-S2 (REIK2)</b>	
<b>Constituents</b>	<b>%</b>	
<b>Aggregate</b>	<b>Inclusions as binder</b>	<b>Inclusions as Aggregate</b>
Quartz	0.7	0.7
Limestone	3.3	3.3
Ironstone	6.2	6.2
Siltstone/Shale	0.5	0.5
Brick	1.1	1.1
Opaque, coal/clinker/ash	25.2	25.2
Lime inclusions	-	15.6
<b>Total Aggregate</b>	<b>37.0</b>	<b>52.6</b>
Binder (Lime)	40.7	40.7
Clinker	1.1	-
Lime inclusions	14.5	-
Secondary products/Calcite and gypsum	6.7	6.7
<b>Total Binder</b>	<b>63.0</b>	<b>47.4</b>
<b>Total Constituents</b>	<b>100.0</b>	<b>100.0</b>
<b>Cracks/Voids</b>	<b>9.6</b>	<b>9.6</b>
<b>Binder: Aggregate Ratio</b>	<b>Total</b>	<b>Effective</b>
	<b>1.0 : 0.6</b>	<b>1.0 : 1.1</b>

**Table No. 3:** Result of modal analysis (600-point count) on thin section RESK1B

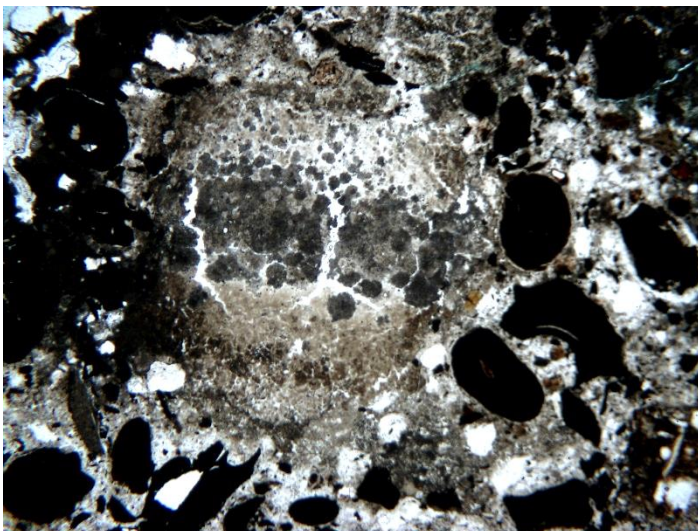


*Photomicrographs:*



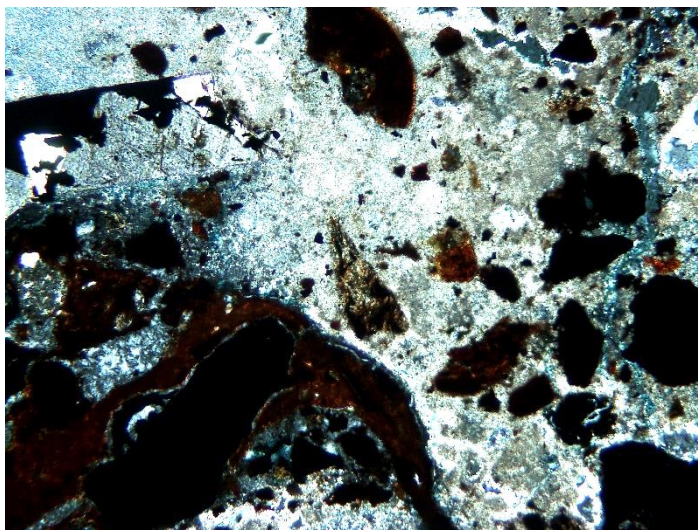
**Plate No. 10:**

A view in plane polarised light (ppl) of an area of the mortar adjacent to the sandstone masonry. A fragment of masonry is shown in the lower right corner. The mortar shows a relatively dense fabric but with a line of small elongated voids (upper part of plate) extending along a fine shrinkage crack extending out from the stone interface. A sulphate filled void can be seen in the lower centre (white). A number of the fine clinker particles display reaction rims and indicate a pozzolanic reaction has occurred. Aggregates are mostly coal and ironstone, with minor quartz. Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.



**Plate No. 11:**

A magnified view in ppl of a large lime inclusion, is shown in the centre of the plate. The inclusion is only partially burnt and partially slaked. The inclusion shows a clear compact margin and it is indicated that it had continued to slake after the mortar was placed. Partial diffusion into the surrounding paste can be seen at the upper part of the plate. Aggregates are dominated by coal fragments, with minor ironstone and clinker. Ash and rare Belite particles can be seen within the paste in the upper right and lower centre and right. Porosity and voids are highlighted by the blue dyed resin. Field of view 2.4mm.



**Plate No. 12:**

A view in Cross Polarised Light (xpl), of an area of paste where depletion of the lime has resulted in an increased microporosity, however, the paste has been heavily altered and is now dominated by gypsum. With the gypsum infilling crack paths, filling voids and diffused through the paste. The aggregates in view are dominated by coal fragments, ironstone with ash and rare clinker particles. The upper left corner shows part of a lime inclusion where the lime has been fully converted. Porosity, the blue impregnating resin and opaque minerals all appear black in xpl. Field of view 1.2mm.

**Rosedale East Iron Kilns**

Examination and Analysis of Mortar samples.



**Summary**

From the examination and analysis of the mortar samples received, from the Rosedale East Iron Kilns, it is confirmed that the mortar used in the fire brick lining and that in the sandstone masonry differ.

Although both appear to have been made from a non-hydraulic air lime, the mortar used to bed the brick was mixed from a hydrate whereas that used in the sandstone masonry was mixed using quicklime as a hot mixed mortar. There is also localised indications to suggest that the mortar used in the sandstone masonry was also placed as a hot mix, in which some late slaking of the lime, whilst confined within the mortar, had occurred.

The mortars both appear to owe their strength to a pozzolanic reaction having occurred between reactive components in the aggregate (coal clinker, ash and iron slag). This is most marked in sample REIK1 from the fire brick lining to the kiln, which was noticeably stronger than the mortar from the Pier masonry. The reactions in the fire brick mortar may also have been aided by exposure to high temperatures in service.

Both mortar samples display the impact of sulphate reactions, with the formation of ettringite and gypsum. The source of the sulphate required for these reactions to occur is likely, at least in part, the aggregate themselves, with e coal clinker and ash a potential source, as is some of the slag, with additional sulphate carried into the mortar in percolating waters, and from exposure to flue gasses from the burning process.

The presence of the high sulphates in the mortar may not impact on the proposed remedial works, 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:

<b>Sample Ref. No.</b>	<b>REIK1</b>	<b>REIK2</b>
Binder form:	Hydrate	Quicklime
Approximate volume proportions calculated on the basis of a Non-Hydraulic lime		
Mix composition by Acid Digestion		
Lime : Aggregate Ratio	1.0 : 0.8	
Mix composition by Modal Analysis		
Lime : Aggregate Total	1.0 : 0.95	1.0 : 0.6
Effective	1.0 : 1.6	1.0 : 1.1

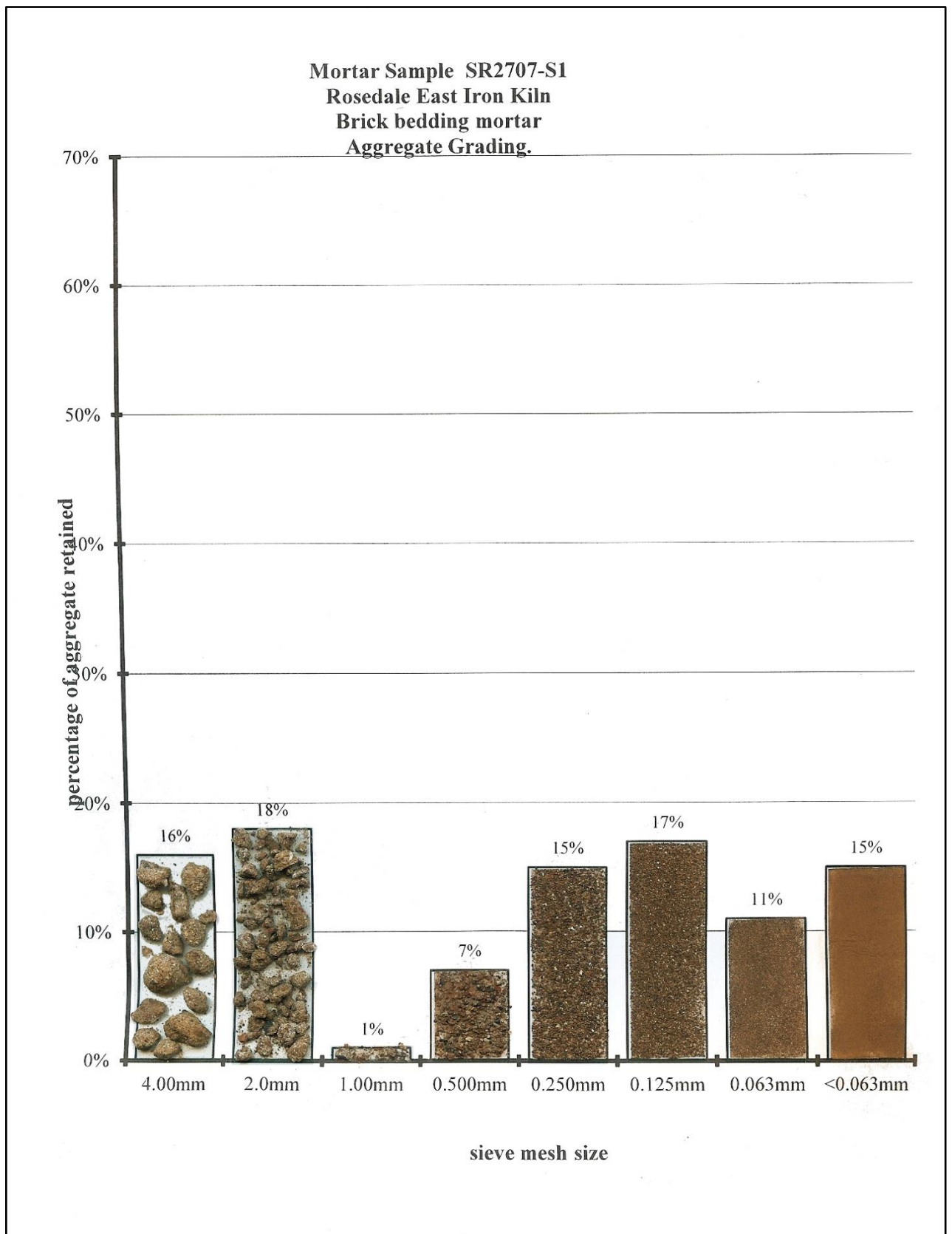
The effective binder content determined from the modal analysis is calculated on the basis that the inclusions are acting as aggregate rather than binder, and this is probably a truer measure of the binder content of the mix, relating to its performance as a mortar. Whereas, the total lime content reflects 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 12<sup>th</sup> March 2019 from Structural & Civil Consultants Ltd., which were identified as mortars from the Rosedale East Iron Kilns, in the North Yorkshire National Park.

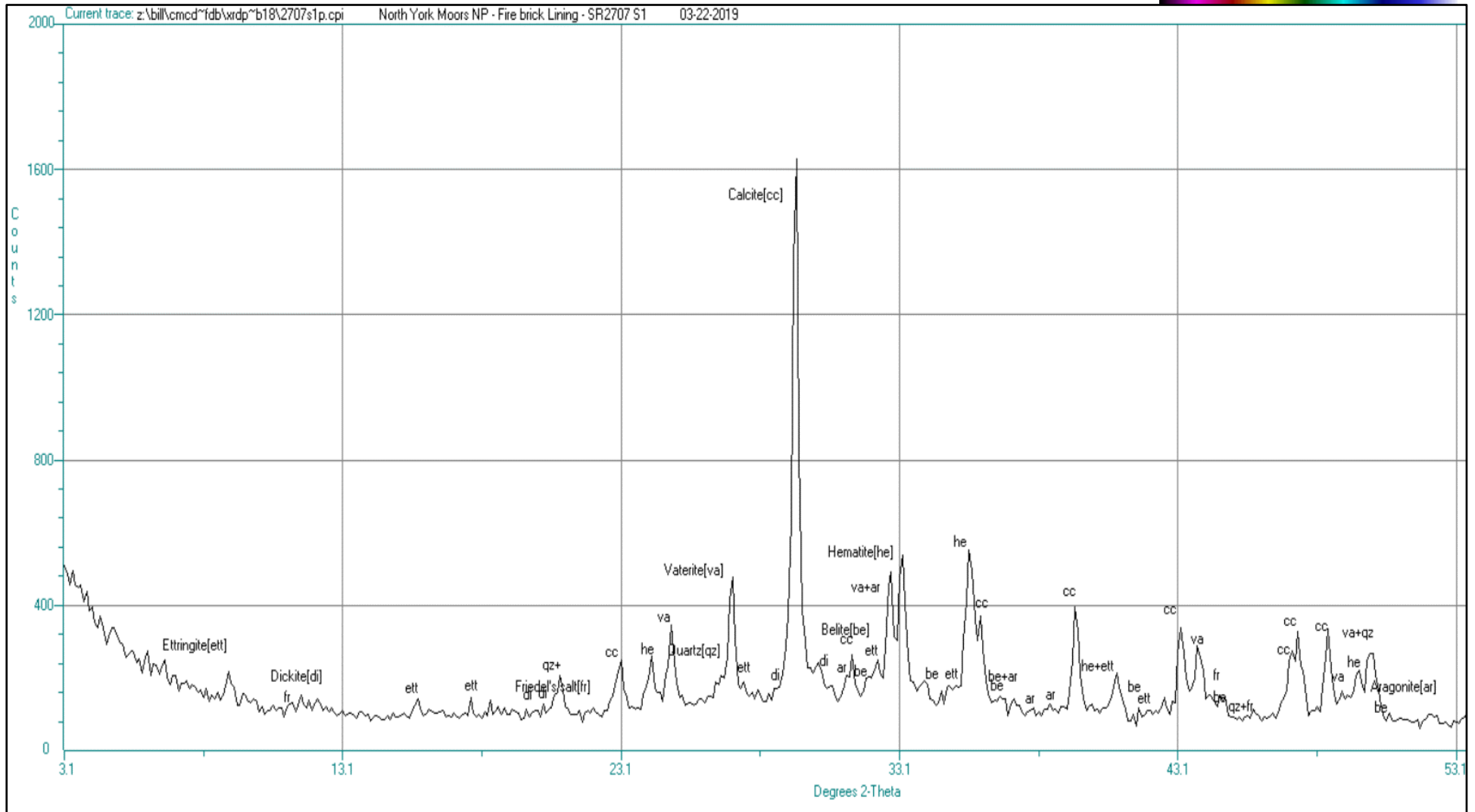




**Figure No. 1:** Aggregate Grading on Aggregate recovered from sample REIK1.

Rosedale East Iron Kilns

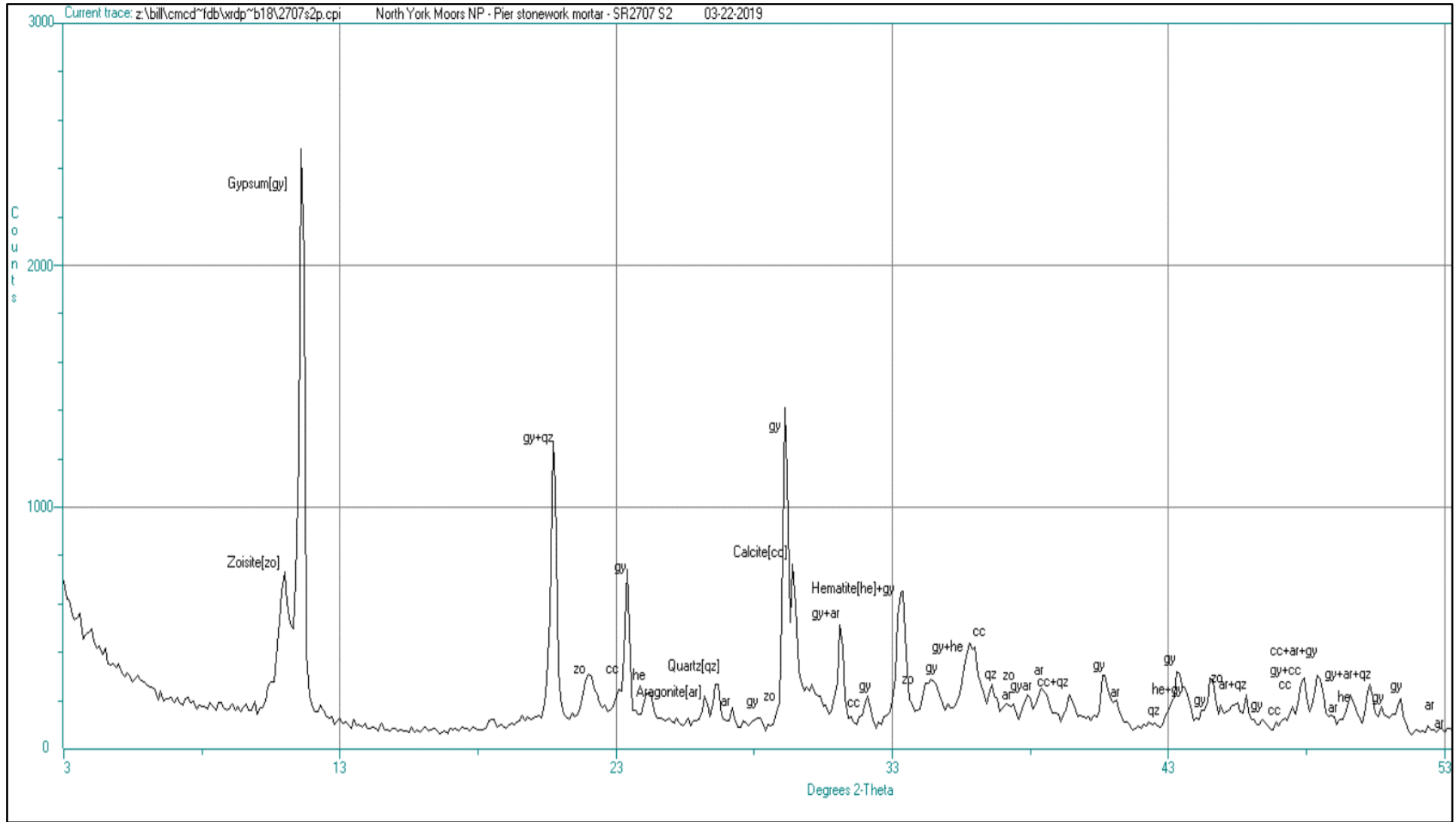
Examination and Analysis of Mortar samples.





**Rosedale East Iron Kilns**

Examination and Analysis of Mortar samples.



**Figure No. 3:** SR2707-S2 (REIK2) Mortar from Pier Stonework – Iron Kiln.