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Our Ref: M/2069/20/C1
Your Ref.: Combs Wood Mine

31st July 2020

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

Project Reference	:	North York Moors NPA – This Exploited Land of Iron
Sample Location	:	Combs Wood Ironstone Mine and Processing Facility, Becks Hole, Goathland, North Yorkshire.
Sample Description	:	Bedding Mortar from Wheel-pit masonry
Date Received	:	29th June 2020
CMC Sample Ref	:	SR 2802-S1
Date Analysed	:	8th to 10th, 21st,23rd and 28th July 2020
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

A mortar sample was received from Nigel Copsey of the Earth Stone & Lime Company, on behalf of the North York Moors National Parks Authority as part of their "This Exploited Land of Iron" project, with a request that the sample be submitted for examination and analysis.

The sample was received in CMC's Stirling Laboratory on the 29th June 2020, and was to be submitted to analysis to determine the composition of the mortar, identification of the binder type along with the grading of aggregates recovered from the mortar.

On receipt in the laboratory, the sample details were entered the sample register and the unique sample identification number SR2802 allocated.

Details of the sample received is given below:

CMC Sample Ref.	Client Ref	Location Sampled
SR2802 – S1	Masonry Mortar	Masonry bedding mortar from a Wheel-pit at Combs Wood Ironstone Mine and Processing Facility.

Method of Test

On receipt in the laboratory the sample was logged, with its mass and size recorded prior to being photographed, in the as-received condition, with the sample submitted to an examination with the aid of a stereo-binocular microscope at magnifications up to x20. During the examination the sample was exposed to a series of *ad hoc* droplet tests employing a range of reagents and indicator solutions. This was to aid identification of the components present in the mortar and to assess their condition.

Following the initial examination, a binder rich sub-sample was obtained from the sample for X-ray Diffraction (XRD) analysis. This was to permit identification of the binder type used in the production of the mortar, indicate if there were hydraulic components present and identify any crystalline reaction products that may also be present.

On the basis of the results from the XRD analysis, a representative sub-sample was prepared for mix composition by acid digestion, following the methods of the Scottish Lime Centre Trust (SLCT). The insoluble residue remaining following the acid digestion 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 to permit clarification of the form in which the binder was used and confirm the mineralogy of the aggregates and other minerals present.

Observations from a Macro/Microscopic examination

On receipt in the laboratory the sample was 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
SR2802-S1	Mortar	1089.9	143.7 x 94.3 x 76.4	5YR 5/3 “Reddish Brown”

The sample consisted of three pieces of mortar all of which were well compacted and the aggregates well bound. The largest piece of mortar adhered to a fragment of a Yellow Brown Sandstone (2.5Y 5/4) to which it was well bonded. The mortar is reddish brown in colour and moderately hard, requiring firm hand pressure to break. Once the intact pieces were broken, the pieces could be disrupted further and powdered under light to moderate finger pressure. It was also noted that although the freshly fractured surfaces were not friable, aggregates could be picked from their surfaces under moderate nail pressure. However, after oven drying, the mortar sample was found to hardened sufficiently to require a hammer impact to disrupt.

On examination under the microscope it was noted that the mortar contained an abundance of dark minerals in the aggregate, with a very few small, sparsely distributed, lime inclusions also observed, these were typically <1.0mm in size and irregular in shape.

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

From a water droplet test, where droplets were applied to both outer surfaces and freshly fractured surfaces, it was indicated that the mortar had a well-connected porosity, with the droplets rapidly absorbed and diffused through the mortar fabric to depth.

Spot tests with dilute Hydrochloric acid produced a strong effervescent reaction, as would be expected on a carbonated lime mortar. A faint odour of hydrogen sulphide (H_2S) was observed on the addition of the acid, indicating that the sample perhaps contained pyrite or other sulphur rich minerals or coal clinker.

Plates No. 1 & 2: The left plate shows the intact pieces in the mortar sample, as-received, note the sandstone fragment adhering to the larger fragment on the right. With the right plate showing a close-up image of a freshly sawn surface cut through the thickness of the sample, after washing to remove cutting dust. Note compact nature of the mortar and the sandstone fragments adhering to the upper surface of the mortar, to which it was very well bonded.

The aggregates appear to be dominated by reddish brown minerals with coal and brick fragments also apparent. With the largest aggregate particle observed being in the region of 3.7mm in size, however, most of the aggregate particles are smaller than 0.5mm.

On testing it was noted that a significant proportion of the aggregates, both coarser than 63 microns, and the fines passing the 63 micron sieve (dust) were weakly attracted to a magnet, indicating the presence of ironstone and/or the presence of a low ferrous slag content.

Results of XRD Analysis for Binder Type

To help clarify the composition of the binder in the mortar, a representative sub-sample was obtained with this lightly crushed in an impact mortar and 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 in the binder, which may only be present in trace proportions.

The powdered sub-sample was back-packed into proprietary sample holder for presentation in the diffractometer, with this technique employed to ensure, as close as possible, the true random orientation of the components present.

The sample was 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 result obtained from the analysis is presented in the following attached Figure, in the form of a labelled X-ray Diffractogram:

Figure No. 2: SR2802-S1 Bedding Mortar from Wheel-pit at Combs Wood Ironstone Mine.

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 and also present with the ironstone,
- **he** = Hematite (Fe_2O_3) Iron oxide, from the ironstone and any slag in the aggregate,
- **go** = Goethite ($FeO(OH)$) Iron oxide hydroxide, weathering product of iron ore,
- **si** = Siderite ($CaFeCO_4$) Iron Carbonate, commonly found in hydrothermal veins and also a common diagenetic mineral in shales and sandstones,
- **al** = Albite, feldspar mineral of the Plagioclase group and Sanidine, a high temperature form of alkali feldspar, present as common rock forming minerals,
- **ill** = Illite, a common clay mineral, similar to mica, present as a rock forming mineral, from the alteration (weathering) of Micas and alkali Feldspars, present within the fines fraction.

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

The results obtained are shown below:

Component	**Proportion (% by Mass)**
Sample:	**SR2802-S1**
Calcite	44.6
Aragonite	2.6
Quartz	16.6
Hematite	21.3
Goethite	1.2
Siderite	1.3
Albite (Feldspar)	6.8
Sanidine (Feldspar)	2.2
Illite	3.4
Total	**100.0**

From the XRD analysis, it is indicated that the mortar is a lime mortar, made with an air lime, a non-hydraulic lime. The strength noted in sample is considered to be due to the presence of a proportion of potentially pozzolanic components in the aggregate.

The high Hematite content confirms the presence of a high proportion of ironstone in the aggregate, this and the presence of Goethite and Illite suggesting the use of mine waste tailings as the aggregate in the mortar production.

Mix Composition

The composition of the mortar was determined by acid digestion, with the result of the analysis carried out presented below:

Sample Ref. No.	**SR2802-S1**	
Binder/Aggregate Ratio	1.0 : 3.4	
Binder form:	Quicklime	Hydrate
Weight proportions calculated mix ratio by dry mass.		
Lime	1.0	1.0
Aggregate	6.0	4.5
Approximate volume Proportions calculated on the basis of a Non-Hydraulic lime		
Lime	1.0	1.0
Aggregate	2.0	1.8

The acid insoluble residue was recovered and this graded through a nest of British Standard Sieves, with the results of the aggregate grading reported below in tabular form, and as an aggregate filled histogram in the appended Figure No. 1.

Sample Reference	**SR2802– S1 Masonry Mortar**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100
4.00mm	0	100
2.00mm	5.3	94.7
1.00mm	7.3	87.4
0.500mm	12.3	75.1
0.250mm	19.5	55.6
0.125mm	28.2	27.4
0.063mm	10.9	16.5
Passing	16.5	

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

The aggregates in the mortar are dominated by opaque minerals along with minor quartz, sandstone fragments, with trace proportions of limestone fragments, indeterminate lithic fragments and brick fragments. The fines fraction contains a low silt/clay content, with most of the fines being composed of quartz, feldspar and hematite.

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 comment on the fabric of the sample, a petrographic thin section was prepared for examination in the polarised light microscope.

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

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

Sample SR2802-S1: Mortar from Wheel-pit, Combs Wood Ironstone Mine

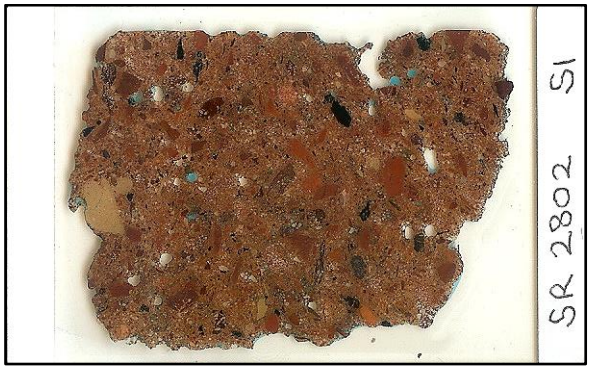

Plate No. 3:

Thin section prepared from mortar sample.

Aggregate

The aggregates in the mortar sample are dominated by ironstone, with minor quartz, feldspar, coal, coal clinker, ash, limestone and brick fragments, along with a trace proportion of clay minerals as coatings on some coarser aggregate particles. The latter possibly derived from alteration (weathering) of the aggregate, and/or present as contamination from soiling. Several small pieces of charred wood are also present.

Ironstone fragments are the dominant component in the aggregate and form approximately 28% of the aggregate, and are present as a mixture of fresh stone and weathered stone fragments, with a low proportion that show indications of having been partially burnt, with these ranging in size from 0.02mm up to 3.6mm.

The coal fragments are a mixture of fresh unburnt particles and partially burnt coal, which along with coal ash clinker, would infer that coal was the fuel used in the iron processing works. The coal fragments range in size from 3.7mm down to 0.08mm. Brick fragments are rare and these have rounded margins along with evidence of weathering, and are typically <0.7mm in size. The brick fragments form a low proportion of the total aggregate and it is unlikely that the brick was added as a pozzolan in the mortar, but rather as a component of the waste material used as aggregate.

Ash is present as fine fragments randomly distributed throughout the paste and as small clusters of poorly bound grains. Ash clinker fragments are also present, up to 2mm in size, but these are rare in the sample examined. A low proportion of limestone fragments were observed, with these mostly angular to sub-angular in shape with both fresh and partially burnt fragments present, they measure up to 2.0mm in size and are rare in the sample.

The aggregates are generally angular to sub-angular, locally rounded to elongate in shape, with a proportion displaying sharp margins and contain cracks, and these may have been crushed. It is, therefore, likely that they had been sourced from the waste material from the processing plant.

Binder

The binder has the appearance of a non-hydraulic lime, with a low proportion of sub-round to irregular shaped lime inclusions observed. Most of the sub-rounded inclusions are fully hydrated, whereas a proportion of the denser fragments appear to be 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 hydrate had the appearance of a powdered hydrate that 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 only minor indications of a patchy reprecipitation within voids that may have contained lime inclusions at an earlier date. Sulphate minerals (ettringite) were rare with these were observed peripheral to coal ash fragments and may infer moisture percolation through the structure sampled, over time.

The lime inclusions range in size from <0.02mm to 1.2mm and have agranular texture, typical of that found in hydrated lime binders, which is likely to suggest, given the age of the mortar, that it had been dry slaked to a hydrate before mixing, either prior to, or mixed, with the aggregate. The binder does, however, appear to have been well slaked. The paste displays a high well connected microporosity, indicating that the mortar was placed as a relatively workable mix.

Voids and microcracks

Voids are mostly irregular to elongated in shape, and are a mixture of placing artefacts with minor dissolution voids, from the depletion of lime inclusions. The voids range in size and shape, from 0.01mm to 2.3mm in size. Most are free of linings albeit rare occurrences of coarse calcite crystals were observed along with rare clusters of sulphate minerals.

Cracks are rare and are random in occurrence and distribution. These are mostly typically of drying shrinkage features.

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

Sample Ref:	**SR2802-S1**	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	11.6	11.6
Sandstone/Siltstone/shale	1.3	2.3
Limestone	2.1	2.0
Opaque: Ironstone	31.6	32.3
Opaque: Coal	9.7	11.2
Coal clinker and ash	4.6	3.0
Brick	3.0	0.3
Wood	0.4	11.6
Lime inclusions	-	1.4
Total Aggregate	**64.3**	**65.7**
Binder (Lime)	33.6	33.6
Lime inclusions	1.4	-
Secondary products/Calcite and sulphate minerals	0.7	0.7
Total Binder	**35.7**	**34.3**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**7.6**	**7.6**
	Total	**Effective**
Binder: Aggregate Ratio	**1.0 : 1.8**	**1.0 : 1.9**

Table No. 2: Result of modal analysis (600-point count) on thin section SR2802-S1

Photomicrographs:

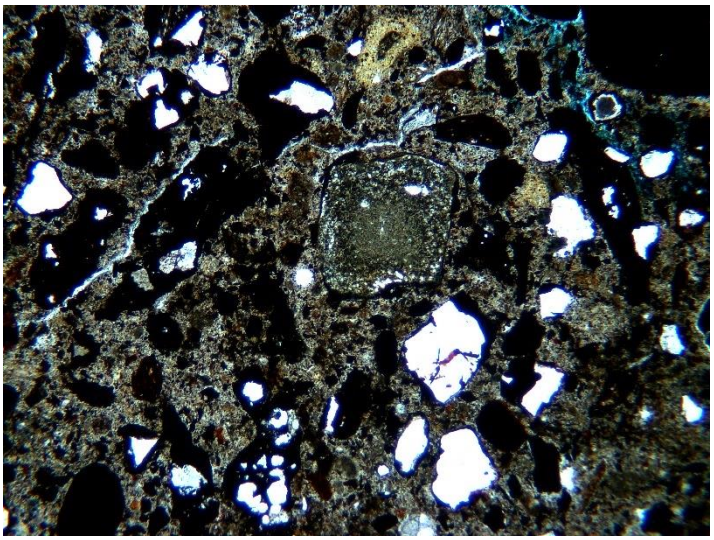

Plate No. 4:

A view in plane polarised light (ppl) of a typical area of the mortar, which shows the aggregates all bound within a dense and fully carbonated paste. The mortar is transected by a series of shrinkage cracks, these are generally free of linings. There is a partially burnt lime inclusion in the centre of the plate, where the encapsulating lime is well diffused into the surrounding paste. The aggregates in view are quartz, ironstone, coal with minor ash and brick fragments.

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

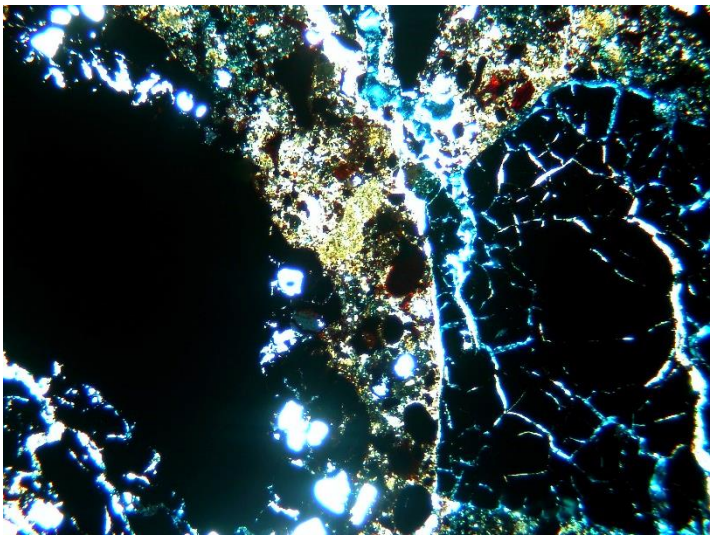

Plate No. 5:

A magnified view in ppl, of an area of mortar containing an ironstone fragment, left side of the plate along with a coal fragment, right side of plate. It is not uncommon to find coal fragments displaying crack patterns of this type within lime rich mortars. The paste contains a crack and an area of high microporosity, upper centre. The ironstone fragment contains fine quartz grains and is bounded by fine ash grains distributed through the paste. Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

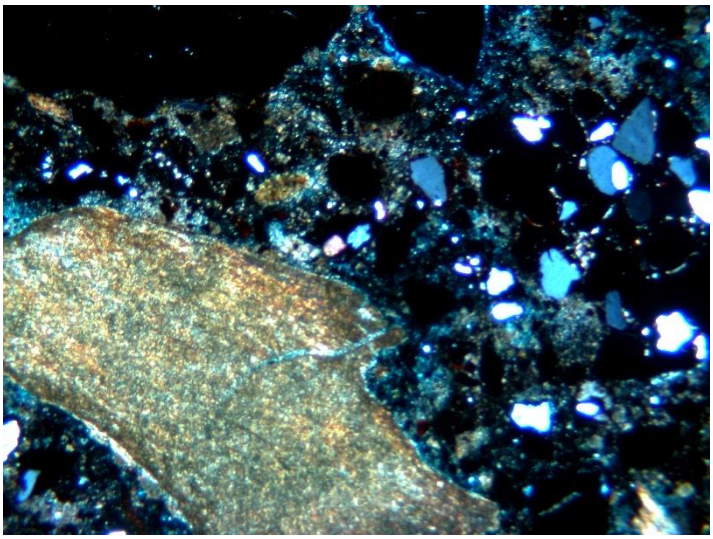

Plate No. 6:

A view in cross polarised light (xpl), of an area of dense fully carbonated paste in which a fresh limestone fragment can be seen, lower left. This is embedded within a carbonated paste along with an ironstone fragment, upper left and lower right, and a coal fragment in the upper right with a quantity of fine quartz grains and ash distributed throughout the paste.

The impregnating resin, porosity and voids all show dark in xpl. Field of view 1.2mm.

Summary

From the examination and analysis of the mortar sample received, from the Bedding Mortar in the Wheel-pit, Combs Wood Ironstone Mine and Processing Facility, Becks Hole, Goathland, North Yorkshire, it is confirmed that the mortar used the masonry was made with a non-hydraulic lime and mine waste as aggregate.

With the mortar mixed using the lime as a quicklime, which appears to have been slaked to a dry hydrate along with the aggregate, although the mortar placed was probably placed as a cold mix.

A summary of the mortar mixes determined is reproduced below:

Sample Ref. No.		**SR2802-S1**
Binder form:		Quicklime
Approximate volume proportions calculated on the basis of a Non-Hydraulic lime		
Mix composition by Acid Digestion		
Lime : Aggregate Ratio		1.0 : 2.0
Mix composition by Modal Analysis		
Lime : Aggregate	Total	1.0 : 1.8
	Effective	1.0 : 1.9

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 sample of mortar received in CMC's laboratory on the 29th June 2020 from the Earth, Stone & Lime Company, which was identified as a bedding mortar from the Wheel-pit at Combs Wood Ironstone Mine and Processing Facility, Beck Hole, Goathland, in the North Yorkshire National Park.

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For CMC Ltd.

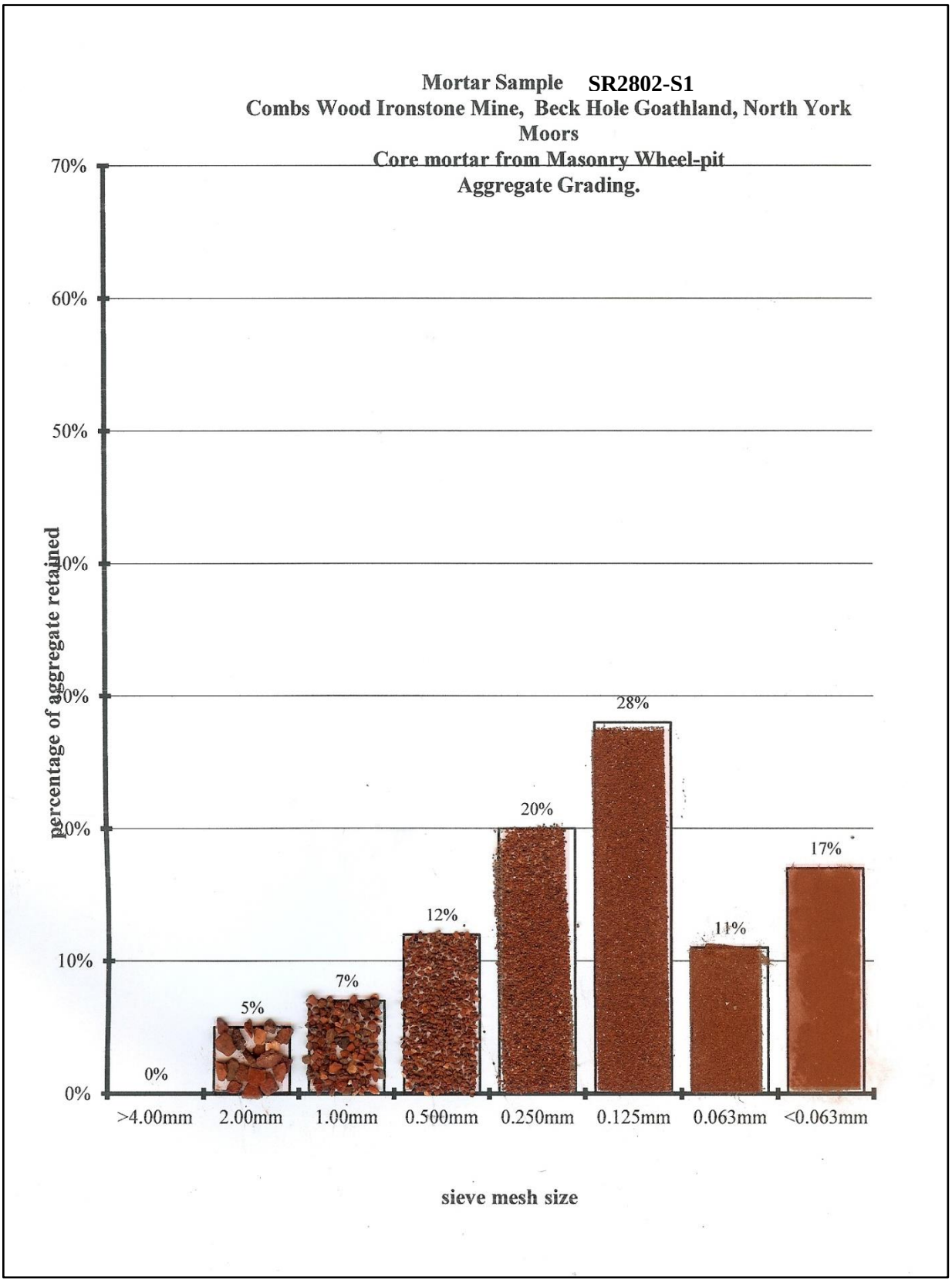

Figure No. 1: Aggregate Grading on Aggregate recovered from sample SR2802-S1.

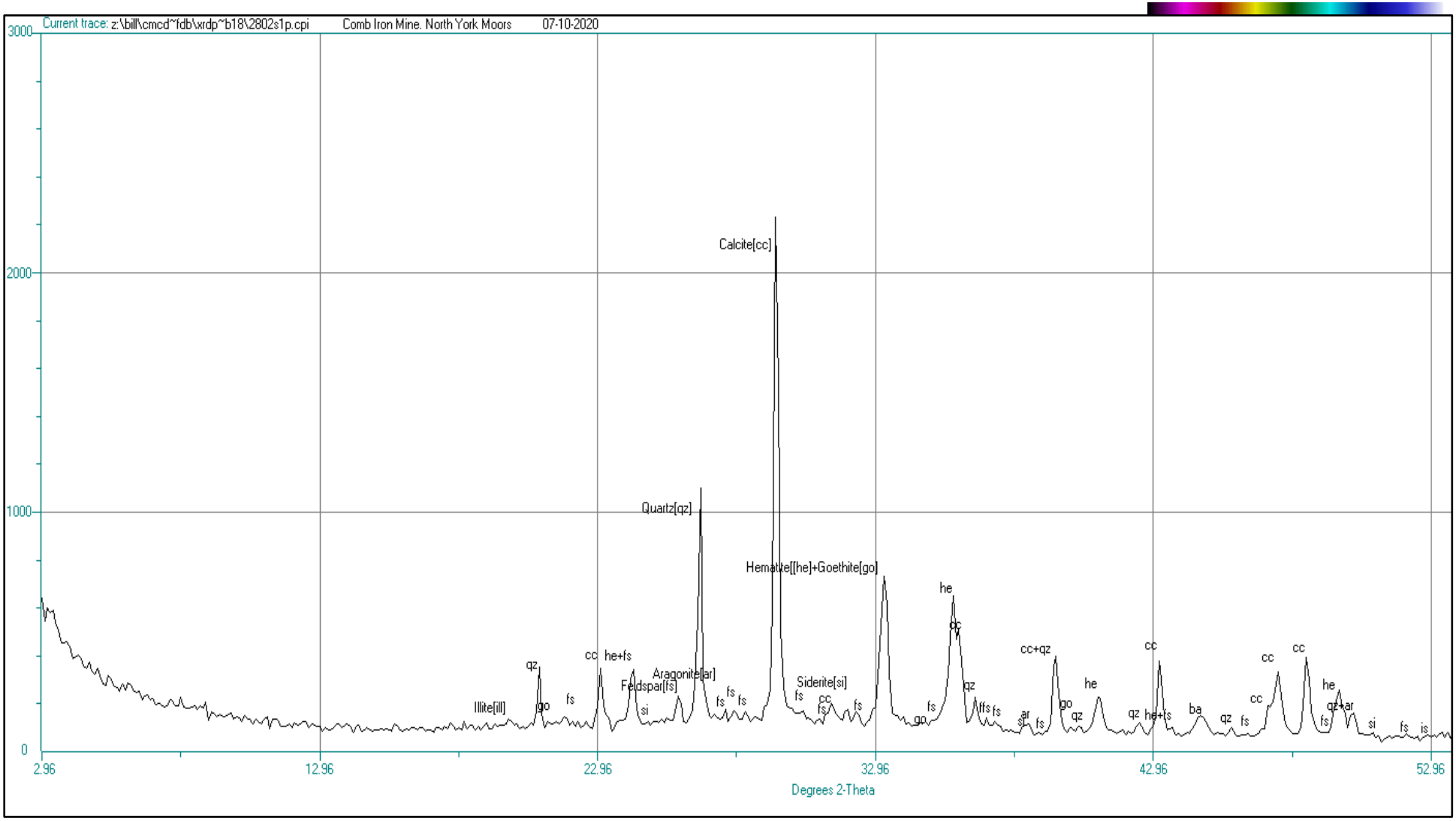

Figure No. 2: SR2802-S1 Bedding from Masonry Mortar in Wheel-pit, Combs Wood Ironstone Mine.