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Our Ref: M/2034/19/C1
Your Ref.: Iron Workers Cottages

7th September 2019

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

Project Reference	:	Iron Workers Cottages, Rosedale
Sample Location	:	Rosedale, North Yorkshire Moors
Sample Description	:	Two coat Plaster from Workers Cottages
Date Received	:	23rd September 2019
CMC Sample Ref	:	SR 2762-S1 Two Coat Plaster
Date Analysed	:	1st to 7th September 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

A sample of plaster was delivered to CMC's Stirling laboratory by Nigel Copsey of the Earth, Stone and Lime Company on the 23rd September 2019. The sample was identified as a two coat plaster from the Iron Workers Cottages in Rosedale, North York Moors.

The plaster was to be submitted to analysis to determine the composition of the plaster in each coat, to include identification of the form and type of binder used in the plaster production. In addition, the grading of the aggregates was to be determined and comment offered on the condition of the plaster as received, and, if possible, on the form in which the binder was used.

On receipt in the laboratory, the sample details were entered the sample register and the unique sample identification number SR2762 allocated.

Details of the sample submitted for examination and analysis is given below:

CMC Sample No.	Description and Location Sampled
SR2762 – S1	Two coat plaster sampled from the walls of the Iron Workers Cottages, Rosedale

Method of Test

On receipt in the laboratory the sample was logged, with its mass and size recorded, with the sample photographed, in the as-received condition.

The sample was then submitted to an examination with the aid of a stereo-binocular microscope at a magnification up to x20 in preparation for analysis, during which each coat in the sample was 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 coat for X-ray Diffraction (XRD) analysis. This was to permit identification of the binder type used in the production of each plaster coat. 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 each coat was prepared for mix composition by acid digestion, following the methods of the Scottish Lime Centre Trust (SLCT). The insoluble residue remaining after 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 aggregates used in each coat to be determined.

In addition to the above a petrographic thin section was prepared, with this orientated to permit both coats to be examined. This permitted clarification of the form in which the binder was used, along with providing confirmation of the mix composition by modal analysis, to permit an allowance to be made for any acid soluble aggregate components present in the plasters.

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
SR2762-S1	Plaster	819.3	190.0 x 108.8 x 47.5	Inner coat 7.5YR 7/2 "Pinkish Grey" Outer coat "White"

This sample consisted of two pieces of a two coat plaster. The two coats were well bonded together and it was necessary to separate them by saw cutting along the interface, to enable samples of each coat to be obtained, without contamination of material from the other coat.

The inner, base coat, plaster ranges from 19mm to 44.3mm in thickness and it is hard and well compacted. The intact pieces could be broken under persistent firm finger pressure, and once broken the base coat could be disrupted further under moderate finger pressure, although the finish coat required impact in a mortar and pestle to disrupt and powder. It was also noted that the freshly fractured surfaces were not readily friable, and the aggregates are well bonded and encapsulated within the paste.

On examination, the base coat plaster was noted to contain an abundance of dark minerals in the aggregate, typical of ironstone, along with possibly some slag material. In addition, there is an abundance of lime inclusions, which measured up to 4.3mm in size, with these observed to be angular and sub-angular to irregular in shape, thereby inferring that the plaster was, perhaps, mixed as a hot mixed mortar, though, perhaps, not applied whilst still hot.

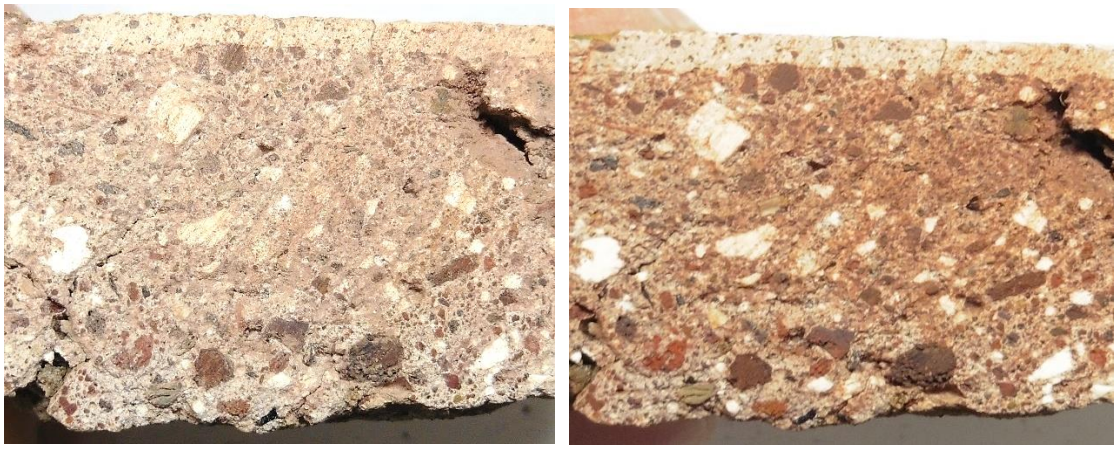
The outer finish surfaces of the plaster is hard and well compacted and was found to range in thickness from 2.6mm to 6.4mm. The aggregates are well bound, and it was only possible to detach aggregates from the paste with the use of a point pick under persistent firm pressure, indicating that the plaster coat was perhaps made from a hydraulic lime, a gauged lime or from a mix which contained a pozzolan.

The surface of the outer finish coat displayed a mapwork pattern of fine cracks, with the cracks highlighted by soiling and algae growths which have become established within them. This would suggest that the sample had been obtained from an area of wall exposed to the environment.

On testing freshly fractured surfaces with a phenolphthalein indicator solution the plaster, in both coats, was found to be fully carbonated.

From the water droplet tests, where droplets were applied to both outer surfaces and freshly fractured surfaces, it was indicated that the plaster displayed a high connected porosity, with the droplets placed onto freshly sawn surfaces and fractured surfaces were rapidly absorbed and diffused throughout the fabric. However, water droplets placed on to the outer surface of the finish coat were supported for some time prior to being absorbed into fine cracks in the surface. This may infer that the plaster had received a surface coating, in the past, or was affected by the soiling and organic colonisation.

Plates No. 1 & 2: The left plate shows the intact pieces in the plaster sample, as-received, with the outer finish coat in view. It was noted that cracks in the finish coat are highlighted by entrapped soiling and algal growths. The right plate shows an internal, masonry contact surface of the inner base coat plaster, with adhered soiling. The soiling is clayey to touch and may infer that the masonry was either bedded in a clay or clay lime mortar, or the interface was exposed to weathering with decay of the plaster at the masonry interface.



Plates No. 3 & 4: The left plate shows a freshly sawn surface through the two coat plaster, where the finish coat, top of plate, is relatively thin and very fine grained. The underlying base coat is coarser grained, with an abundance of lime inclusions apparent, along with ironstone aggregates. The right plate shows the sample after wetting to highlight the two coats, during which it was noted that the base coat appeared more red in the moist condition, as did the ironstone aggregates.

Spot tests with dilute Hydrochloric acid produced a strong effervescent reaction, as would be expected on a carbonated lime plasters/mortar, although, it was noted that the reaction was accompanied by a weak smell of hydrogen sulphide (H_2S), which perhaps indicates that the sample contained pyrite or other sulphur rich minerals within the paste or the aggregates.

The aggregates appear to be dominated by dark minerals with ironstone, coal, clinker and brick fragments all apparent. With the largest aggregate particle observed being in the region of 6.2mm in size, however, most of the aggregate particles are finer than 1.0mm.

Results of XRD Analysis for Binder Type

To help clarify the composition of the binder in the plaster, a representative sub-sample was obtained from each coat. These were 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 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. The 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. **3**: SR2762-S1A - Inner base coat plaster ex Iron Workers Cottage,

Figure No. **4**: SR2762-S1B - Outer finish coat plaster ex Iron Workers Cottage.

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, also found in some forms of redeposited leached lime binder,
- **qz** = Quartz (SiO_2) Silicon Oxide, a component of the aggregate and also in some of the slag fragments,
- **be** = Belite (C_2SiO_4) *di*-Calcium Silicate, clinker component in binder and occasionally found in slags and in ashes, and other pozzolanic materials,
- **fr** = Friedel's Salt ($Ca_4Al_2O_6Cl_210H_2O$) Calcium Aluminium Oxide Chloride Hydrate, hydration product, from the hydration of clinker and in some pozzolanic reactions
- **hd** = Hydromagnesite ($Mg_5(CO_3)_4(OH)_24H_2O$) Magnesium Carbonate Hydroxide Hydrate, component of the binder, or from the slag/pozzolan in the aggregate,
- **fe** = Iron (Fe) metallic iron, found within some slag fragments, included as aggregate,
- **he** = Hematite (Fe_2O_3) Iron oxide, from the ironstone and some of the slag included in the aggregate,
- **si** = Siderite ($FeCO_3$) Iron Carbonate, present in the slag/aggregate, and possibly as a reaction product,
- **fs** = Feldspar, mostly Albite and Anorthite of the Plagioclase group of minerals, aggregate component,
- **mi** = Muscovite mica, common rock forming mineral, aggregate/clay component.

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	**Proportion (% by Mass)**	
Sample:	**SR2762-S1A**	**SR2762-S1B**
Coat	**Base Coat**	**Finish Coat**
Calcite	69.6	69.4
Aragonite	8.1	2.1
Quartz	0.5	8.0
Belite (B_2S)	3.3	1.7
Friedel's Salt	1.5	2.2
Iron	0.7	5.8
Hematite	12.7	2.9
Siderite	2.0	2.7
Feldspar (Albite)	0.1	0.2
Feldspar (Anorthite)	1.3	2.2
Muscovite Mica	0.2	0.6
Hydromagnesite		2.2
Total	**100.0**	**100.0**

From the XRD analysis, it is indicated that the plaster was made with a high calcium lime, with the hydraulicity observed originating from the pozzolanic effect of the slag and ashes added to the plasters, in both coats.

The Hematite and metallic Iron content found, in both samples, is a function of the high proportion of ironstone and slag material within the aggregates incorporated in the plaster coats.

Mix Composition

The composition analysis was determined by acid digestion, following the procedures of the SLCT. The results from the analysis carried out are presented below:

Sample Ref. No.	**SR2762-S1A**	**SR2762-S1B**
Coat	**Base Coat**	**Finish Coat**
Mortar type (from XRD)	Non-Hydraulic Lime	Non–Hydraulic Lime
Binder/Aggregate Ratio	1.0 : 0.9	1.0 : 0.2
Binder form:	Quicklime	Putty
Weight proportions calculated mix ratio by dry mass.		
Lime	1.0	1.0
Aggregate	1.7	0.2
Approximate volume Proportions calculated on the basis of a Non-Hydraulic lime		
Lime	1.0	1.0
Aggregate	0.7	0.14

The residue from the acid digestions were recovered and the particle size distribution determined on both samples. With the result of the grading analysis presented in the following table, along with the results presented in the form of aggregate filled histograms, in the appended Figures No. 1 and 2.

Sample Reference	SR2762– S1A Base Coat Plaster		SR2762– S1B Finish Coat Plaster	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100	0	100
4.00mm	1.2	98.8	0	100
2.00mm	11.9	86.9	0	100
1.00mm	15.3	71.6	0	100
0.500mm	9.5	62.1	3.4	96.6
0.250mm	21.4	40.7	11.8	84.8
0.125mm	16.8	23.9	22.7	62.1
0.063mm	9.4	14.5	19.7	42.4
Passing	14.5		42.4	

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

The aggregates in the mortar are dominated by opaque minerals (including ironstone and coal) along with slag, coal ash and limestone fragments with trace proportions of quartz and indeterminate lithic fragments, with feldspars and muscovite mica in 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 rare 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 coats, a petrographic thin section was prepared for examination in the polarised light microscope. To achieve this a slice was sawn through an intact piece of the plaster, which was orientated to include both coats in the one section. The slice was dried at 70°C prior to being impregnated with a blue dyed epoxy resin, in preparation for the manufacture of a thin section.

One side of the impregnated sample was cut and polished prior to being mounted on to a glass slide (50mm x 75mm), with the sample orientated to give the maximum area on the slide. The sample was then cut and polished to give a thickness in the region of 30µm, prior to being protected by a cover slip, in preparation for examination in the polarised light microscope.

The thin section was examined in an Olympus BH2 Polarised light microscope, which was fitted with a digital camera to permit the recording of images for record purposes. A selection of the images are included in the report for reference.

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

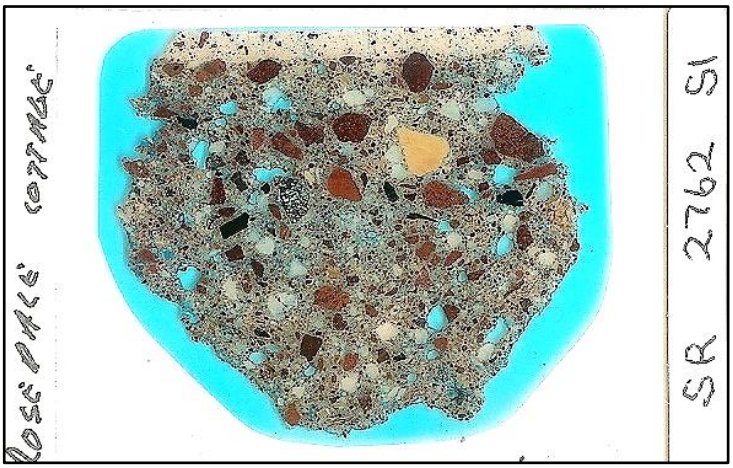


Plate No. 5:

Thin section prepared from Plaster sample SR2762-S1.

The outer finish coat is seen at the top of the plate.

Note the high ironstone content in the base coat plaster.

Aggregate

Base Coat Plaster

The aggregates in the plaster sample are dominated by ironstone, coal, coal clinker, ash, along with minor limestone and fine sandstone/siltstone fragments, and rare small brick fragment. In addition, there are trace proportion of quartz, along with feldspars and mica in the silt fraction.

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 3.8mm down to <0.05mm. Brick fragments are rare; these are small and have rounded margins with evidence of weathering and are typically <0.2mm 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 6.2mm to <0.02mm.

Ash is present both in the form of clinker and as fine fragments diffused through the paste and locally occurs as small clusters of poorly bound grains. A low proportion of limestone fragments were observed, with these angular to sub-angular in shape with no evidence of having been burnt and are therefore considered to have been included along with the aggregate, they measure up to 0.54mm in size, but mostly <0.05mm in size.

Although the aggregates are dominated by ironstone, slag, coal and ash there is a minor proportion of natural aggregate present. The latter appear to be generally sub-angular to sub-round, to elongate in shape and less than 0.1mm in size. The coarser fragments contain a high proportion of particles displaying sharp margins and these have the appearance of having been crushed. It is, therefore, likely that the source of the aggregates used in the plaster was the waste material product from the Iron processing Kilns.

Finish Coat Plaster

The aggregates in this coat appear to have been screened from that used in the base coat and are dominated by ironstone, along with quartz and coal fragments, with only trace proportions of other naturally occurring aggregate minerals observed.

The particles are typically angular to sub-angular in shape, with a maximum size of 0.64mm, but mostly finer than 0.1mm.

Binder

Base Coat Plaster

The binder has the appearance of a non-hydraulic lime, with a high proportion of sub-angular to irregular lime inclusions observed. The lime inclusions are variably slaked, with a low abundance of incompletely slaked lime, along with a few displaying overburnt outer margins. However, most are fully calcined, although a proportion of these are incompletely slaked.

Locally sub-rounded inclusions are fully hydrated and have the appearance of a putty. However, there is also a significant proportion of the denser fragments which are 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 quicklime, although given the intimate contact with the paste and the absence of voids and other features consistent with hot mixed and placed mortars, it is considered that the base coat plaster, albeit mixed as a hot mixed mortar, was allowed to cool and had been remixed prior to placing.

The paste is fully carbonated throughout the sample, and within the base coat there is little evidence of leaching or the redeposition of calcite.

The inclusions range in size from <0.02mm to 2.8mm and it is inferred from the examination of these, that the limestone burnt was calcareous and mostly micritic to crystalline in form, albeit there are minor indications that a proportion of the limestone was bioclastic/Oolitic.

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, as there was no clinker apparent within the lime inclusions, it was considered that the hydraulic components had originated from another source, other than the lime. Therefore, it is concluded that the pozzolanic materials (coal clinker, ash and slag from iron processing), a proportion of which displays reaction rims, originated within the waste material from the iron processing operations, used as the aggregate.

Finish Coat Plaster

The binder in the finish coat is dense and well compacted, it has the appearance of having been mixed from a putty lime, with small putty inclusions distributed throughout. The putty is likely to have been prepared from the quicklime used in the base coat plaster.

The binder although dense is fully carbonated and is locally transected by shrinkage cracks extending from the outer surface to the interface with the base coat, however the cracks do not penetrate the base coat plaster and the bond between the finish and base coats is well formed and intact.

Locally leaching of binder has occurred at crack margins with localised redeposition within the crack pathways, but this is minimal. Evidence of a thin, partially absorbed surface coating is apparent, in patches across the outer surface of the finish coat. This perhaps suggesting the application of a limewash, or distemper, coating applied as a decoration.

Voids and microcracks

The voids observed in the base coat plaster 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 in the base coat range in size and shape, with voids ranging from 0.04mm to 2.3mm in size. Some of the voids retain a partial coating of disrupted lime from depleted inclusions whilst the cracks in the finish coat locally retain coarse calcite crystals.

Cracks are rare in the base coat but are common in the finish coat. Those in the base coat are fine shrinkage cracks typically <0.02mm in width, they are random in occurrence and were observed around the perimeter of some lime inclusions and skirting coarser aggregate particles and locally connecting larger slag particles. The cracks in the finish coat are typical of early drying shrinkage features. They extend through the full thickness of the finish coat, from the outer surface and range from <0.01mm to 0.3mm in width. The cracks are commonly lined with secondary mineral deposits, dominated by calcite.

Sample Ref:	SR2762-S1A Base Coat Plaster		SR2762-S1B Finish Coat Plaster	
Constituents	**%**		**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	1.9	1.9	1.8	1.8
Limestone	1.3	1.3	0	0
Lithic	0.7	0.7	0.1	0.1
Sandstone/Siltstone/shale	1.7	1.7	0	0
Brick	0.1	0.1	0	0
Opaque, Ironstone	29.7	29.7	10.8	10.8
Opaque, coal	9.1	9.1	1.3	1.3
Wood fragments	0.1	0.1	0	0
Lime inclusions	-	16.7	-	22.2
Total Aggregate	**44.6**	**61.3**	**14.0**	**36.2**
Binder (Lime)	38.5	38.5	62.5	62.5
Lime Inclusions	12.3	-	15.2	-
Clinker & Ash	4.4	-	7.0	-
Secondary products - Calcite	0.2	0.2	1.3	1.3
Total Binder	**55.4**	**38.7**	**86.0**	**63.8**
Total Constituents	**100.0**	**100.0**	**100.0**	**100.0**
Cracks/Voids	**7.3**	**7.3**	**8.2**	**8.2**
	Total	**Effective**	**Total**	**Effective**
Binder: Aggregate Ratio	**1.0 : 0.8**	**1.0 : 1.63**	**1.0 : 0.16**	**1.0 : 0.56**

Table No. 2: Result of modal analysis (600-point count) on thin section from SR2762-S1.

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 that the ash was added as aggregate rather than with the lime, which 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.

Photomicrographs:

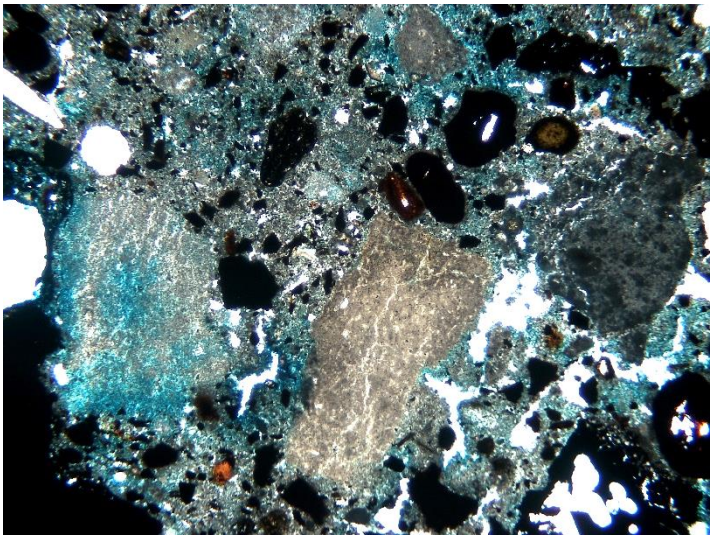


Plate No. 6 - Base Coat Plaster:

A view in plane polarised light (ppl) of a typical area of the mortar, which shows the aggregates encapsulated in the paste. There are three lime inclusions apparent in this view, with that on the lower left being well calcined but poorly hydrated, with the lower centre inclusion being incompletely calcined and retains part of the original limestone fabric, whereas that in the centre right is overburnt and had not slaked. The aggregates in view are dominated by ironstone fragments. The paste in the lower right is partially depleted, whereas that in upper left is compact. Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

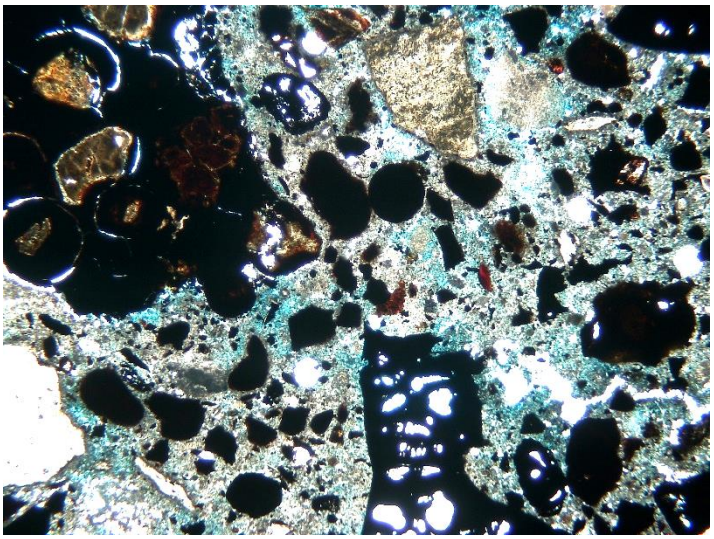


Plate No. 7 – Base Coat Plaster:

A magnified view in Cross polarised light, xpl, of an area of paste where the paste is locally depleted, upper right, with a patchy high microporosity also noted. The aggregates in view are dominated by ironstone fragments, see upper left, along with coal and partially burnt coal clinker, bottom centre. Narrow reaction rims can be seen around small fragments of clinker and some fine ironstone fragments. The cracks in view are free from secondary mineral deposition. The paste is fully carbonated and free of any evidence of deleterious reactions. The blue dyed resin, porosity and voids all appear dark in xpl. Field of view 2.4mm.

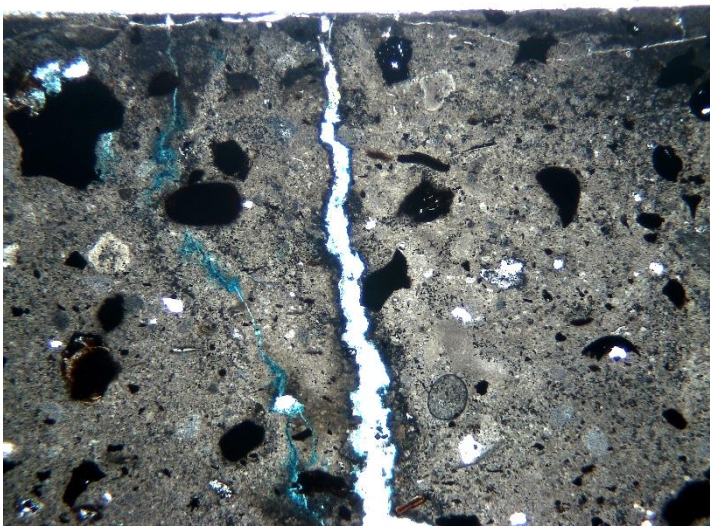


Plate No. 8 – Finish Coat Plaster:

A view in plane polarised light (ppl), of the near surface of the finish coat plaster, with the outer surface at the top of the plate. A shrinkage crack widens towards the inner contact surface, and is a common feature, indication water loss into the base coat when placed. Aggregates are dominated by ironstone, with minor coal fragments and fine quartz grains. Small patches of lime putty can be discerned within the paste. The crack margins are coated in redeposited calcite. The paste is dense and fully carbonated. Porosity is highlighted by the blue dyed resin. Field of view 2.4mm.

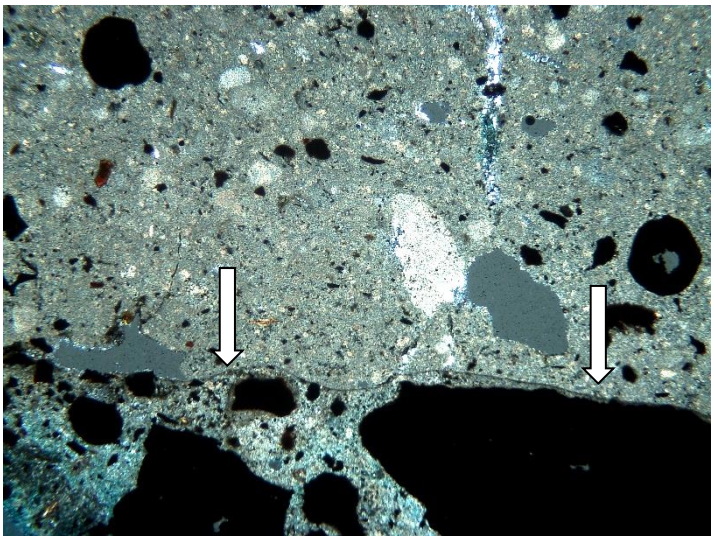


Plate No. 9:

A view in cross polarised light (xpl), of an area of the finish coat at its interface with the base coat plaster. Base coat is at the bottom of the plate, with the interface arrowed in plate. The paste of the finish coat is dense and fully carbonated and displays a low aggregate content, with the aggregate in view dominated by ironstone fragments and coal, along with fine quartz grains and ash. Two elongated air voids (grey) can be seen towards the base coat interface, formed from entrapped air during placing. The impregnating resin, porosity and voids all show dark in xpl. Field of view 2.4mm.

Summary

From the examination and analysis of the plaster sample received, from the Iron Workers Cottages at Rosedale in the North York Moors, it is confirmed that the plaster in both coats was based on a non-hydraulic air lime. However, although both appear to have been made from a non-hydraulic air lime, the mortar used in the base coat plaster was mixed using the lime in the form of a quicklime, whereas that used in the finishing coat plaster was mixed with a lime in the form of a putty lime.

The plaster in both coats appear to have gained their strength due to a pozzolanic reaction having occurred between reactive components in the aggregate (coal clinker, ash and iron slag).

A summary of the mortar mixes determined is reproduced below:

Sample Ref. No.		**SR2762-S1A**	**SR2762-S1B**
Plaster Coat		**Base**	**Finish**
Binder form:		Quicklime	Putty Lime
Approximate volume proportions calculated on the basis of a Non-Hydraulic lime			
Mix composition by Acid Digestion			
Lime : Aggregate Ratio		1.0 : 0.7	1.0 : 0.14
Mix composition by Modal Analysis			
Lime : Aggregate	Total	1.0 : 0.8	1.0 : 0.16
	Effective	1.0 : 1.63	1.0 : 0.56

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 two coat Plaster received in CMC's laboratory on the 23rd September 2019 from the Earth, Stone & Lime Company, which was identified as plaster from the Iron Workers Cottages in Rosedale, North Yorkshire Moors.

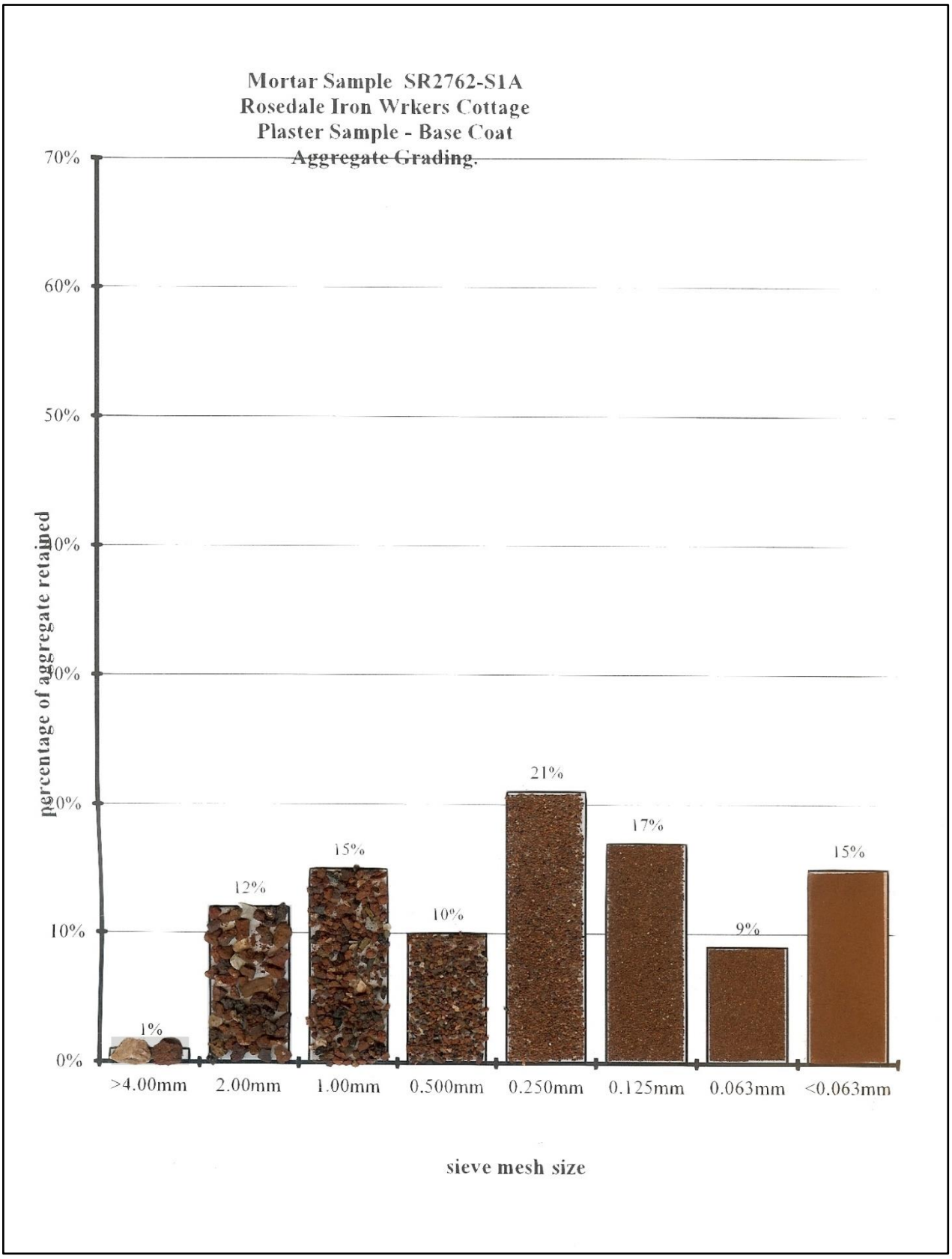


Figure No. 1: Aggregate Grading on Aggregate recovered from Base Coat Plaster.

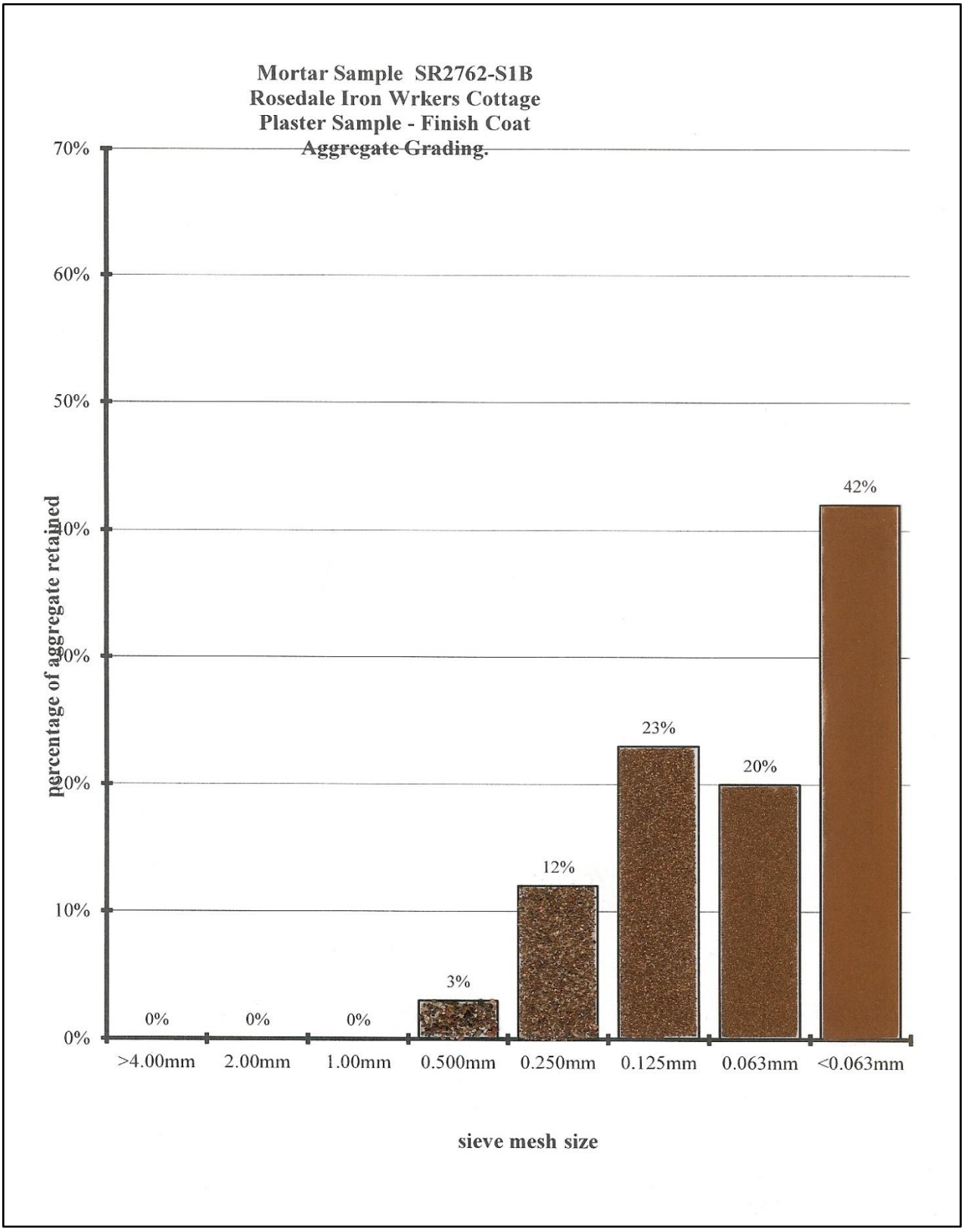


Figure No. 2: Aggregate Grading on Aggregate recovered from Finish Coat Plaster.

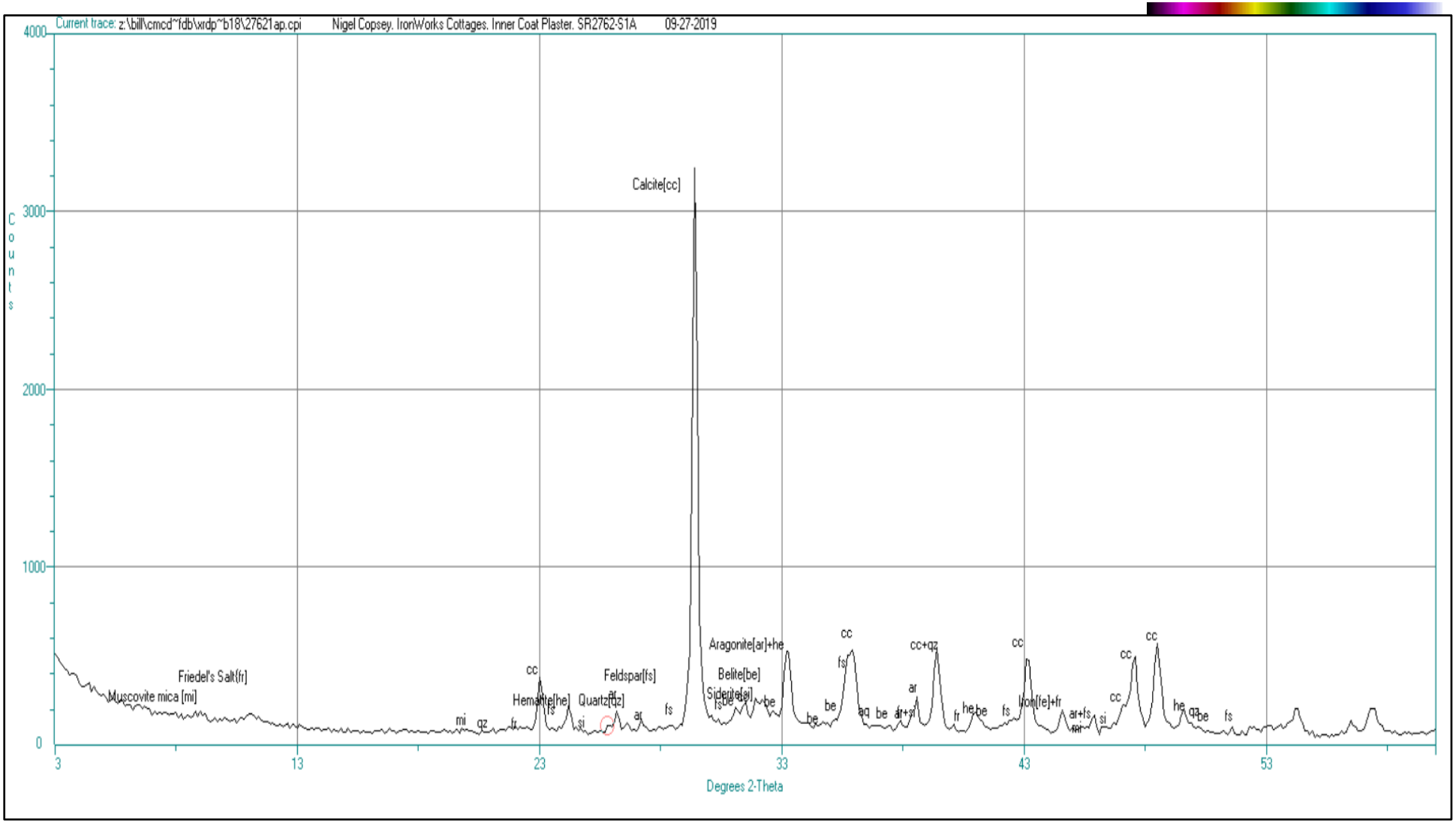


Figure No. 3: SR2762-S1A - Inner base coat plaster ex Iron Workers Cottage.

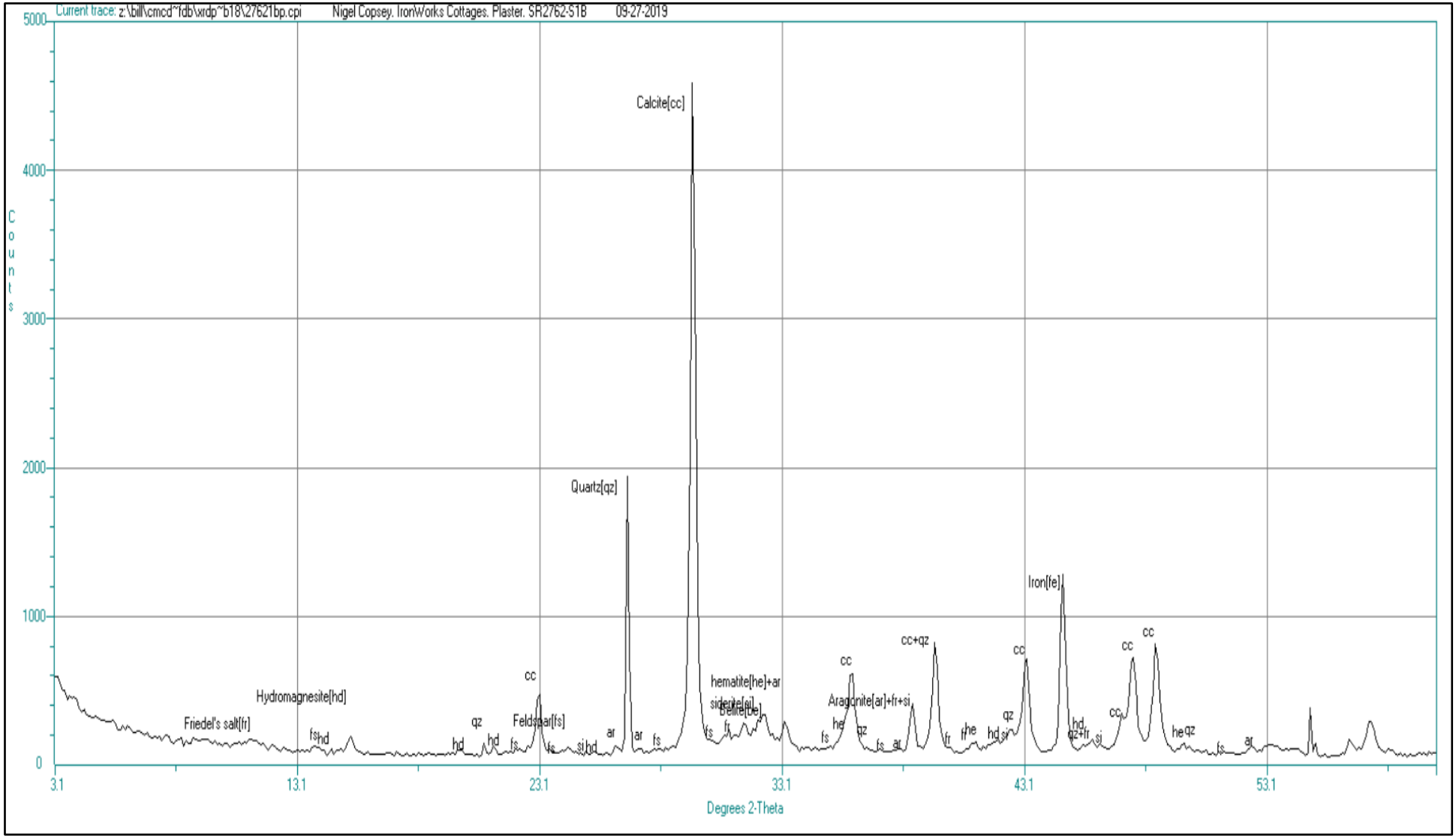


Figure No. 4: SR2762-S1B - Outer finish coat plaster ex Iron Workers Cottage.