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Our Ref: M/2013/19/C1
Your Ref.: Old York House

11th September 2019

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

Project Reference	:	Old York House, Malton, North Yorkshire
Sample Source	:	Internal Plasterwork
Sample Description	:	Finish coat Plaster.
Date Received	:	19th June 2019
CMC Sample Ref	:	SR 2735 - S1
Method of Test	:	Mix composition by acid digestion, and binder type by XRD analysis by in-house procedures, with fabric condition from a thin section examination.

Samples

A sample of plaster was received from Nigel Copsey of the Earth, Stone & Lime Company in CMC's Stirling laboratory on the 19th June 2019. The sample was identified as "Finish Coat Plaster" placed over a clay mortar basecoat, in the Old York House, Malton, North Yorkshire.

The purpose of having the sample analysed was to obtain a detailed understanding of the mortar mix used in the production of the plaster, including mix composition, binder type and form in which used, with comment on the hair reinforcement within the plaster coat.

On receipt in the laboratory, the sample details were entered into the sample register and the unique sample identification number SR2735 allocated. The details of the sample received is presented below:

CMC Ref.	Client Ref	Location Sampled
SR2735 - S1	Plaster	Plaster from internal walls, over clay mortar, in the Old York House, Malton.

Method of Test

The sample was initially photographed on receipt in the laboratory, logged with its mass and size recorded prior to being submitted to an examination with the aid of a stereo-binocular microscope at a magnification 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 to aid the identification of the components present and to assess the condition of the plaster as received.

To permit confirmation of the type of binder used in the production of the plaster a representative sub-sample was prepared and this and analysed by X-ray Diffraction (XRD). To achieve this the representative sub-sample was initially disaggregated by gently grinding it in an agate mortar and pestle to separate the binder from the aggregates and the hair, with a binder rich sub-sample recovered by sieving the disaggregated material over a 63µm sieve.

The prepared powdered sample, passing the sieve was backpacked into a proprietary sample holder in preparation for presentation in the diffractometer. The sample was analysed in a Philips X-ray Diffractometer fitted with a single crystal monochromator, set to run over the range 3° to 60° 2θ in steps of 0.1° 2θ at a rate of 1° 2θ/minute using CuKα radiation. With the digital output from the diffractometer analysed 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°.

Following the initial examination, a further representative sub-sample sample was prepared, with this dried to a constant weight and the materials disaggregated in a mortar and pestle in preparation for digestion of the binder in dilute hydrochloric acid. The acid digestion was carried out following the procedures of the Scottish Lime Centre Trust (SLCT). On completion of the acid digestion the aggregates were recovered by vacuum filtration, dried and then graded through a nest of Standard sieves.

In addition to the above an intact piece from the sample was selected for the preparation of a petrographic thin section. This to be used to assist in establishing the form in which the binder was used in the production of the plaster.

Observations from Macro/microscopic examination

The sample was logged on receipt with the following determined:

Sample Ref.	Client Ref.	Mass of Sample (gram)	Dimensions of Largest piece (mm)	No. of Pieces	Colour by the Munsell Colour Charts
SR2735-S1	Plaster	99.2	155.0 x 91.6 x 10.6	3 No	"White"

This sample consisted of three pieces of a lime plaster. The plaster was observed to be well compacted but contain an abundance of cracks, however, it was held together by an abundance of hair reinforcement, which appeared to be in a good condition. The plaster was firm but friable and pieces could be broken under moderate to light finger pressure and once disrupted the pieces could be powdered under light to moderate finger pressure, and aggregates could be picked from the mortar under light to moderate nail pressure.

On testing a freshly fractured surface with a phenolphthalein indicator solution the plaster was found to be fully carbonated. Water droplet tests placed onto the inner, clay mortar, contact surface and on freshly fractured edges confirmed that the plaster contained a well connected pore structure, with the droplets, placed absorbed very quickly and diffused through the full thickness of the pieces tested. However, droplets placed onto the outer surface were supported for an extended period prior to slowly being absorbed. This may infer that the outer surface had retained a decorative finish, a lime wash, distemper or similar form of coating.

The outer surface of the plaster was noted to be particularly dense and appeared to retain a thin coating. On measurement this was found to range from 0.34mm to 0.65mm in thickness, and the coating was less friable than the material within the thickness of the finish coat plaster. On examination it was noted that the surface was well pressed back and deficient in aggregate and had the appearance of a putty lime skim coat, over which a thin layer, <0.1.5mm thick, coating had been applied, possibly distemper or limewash.


Plates No. 1 & 2: The above plates show the condition of the sample as received, with the left plate showing the outer coated surface of the individual pieces, as received. The right plate shows a magnified view the inner clay mortar contact surface, with the plaster retaining patches of clay and some pieces of straw, presumably from the clay plaster base coat.


Plates No. 3 & 4: The left plate above is a close-up of a fractured edge, showing the abundance of hair present in the plaster coat. With the right plate being a magnified view of the inner contact surface with retained clay mortar and straw reinforcement from the base coat plaster adhering strongly to the plaster finish coat.

Plate No. 5:

The plate on the left is a close-up of the surface, showing the thin outer surface formed from a skim coat, which had received a surface applied coating at some time in the past. The coating may be a distemper or a lime wash, but to confirm if it was a gypsum gauged skim coat a sample was obtained and analysed by XRD.

The aggregates within the body of the plaster coat had the appearance of fine quartz grains along with limestone, with locally small darker minerals that may infer a proportion of coal or coal ash clinker in the coating. There was also a quantity of lime inclusions observed within the plaster coat, with these being mostly sub-rounded in shape and they may have formed from incompletely mixed putty lime.

Results of XRD Analysis

To confirm if the binder was a lime, and determine if it was hydraulic, or not, a binder rich sub-sample was obtained and submitted to analysis by X-ray Diffraction (XRD). In addition, a sample of the outer surface was also obtained, and this prepared for analysis separately.

The results of the analysis are presented in the following Figure, in the form of labelled X-ray Diffractogram:

Figure No. 2: Sample SR2735-S1 – Internal Finish Coat Plaster, ex The Old York House, Malton,

Figure No. 3: Sample SR2735-S1a – Thin coating on outer surface of Finish Coat Plaster

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

- **cc** = Calcite ($CaCO_3$) calcium carbonate, carbonated binder from lime binder, and also any limestone present in the aggregate,
- **ar** = Aragonite ($CaCO_3$) another crystalline form of calcium carbonate, carbonated lime binder, also, a common component of shell and found in limestone, may also be present from any applied surface coating (decoration) i.e. lime wash/paint, or whitening (limestone dust/chalk) in distemper,
- **qz** = Quartz (SiO_2) natural aggregate component present as an aggregate or a contaminant.
- **gy** = Gypsum ($CaSO_4 2H_2O$) Calcium Sulphate hydrate, Present as an environmental sulphate reaction product, as its concentration is too low to suggest that it was added to the plaster,
- **br** = Brownmillerite ($Ca_2(Al, Fe)_2O_5$) Calcium Aluminium Iron Oxide, Hydraulic/Pozzolanic component, most likely present as a component of the ash in the plaster coat,
- **he** = Hematite (Fe_2O_3) Iron Oxide, iron ore mineral in the aggregate, or a contaminant in the ash observed within the plaster coat.

On the basis of the XRD analysis, it is indicated that plaster sample was made from a non-hydraulic air lime. The plaster is fully carbonated and there was only limited evidence of a sulphate reaction product present, which may be due to the presence of ash in the mix, or the age of the plaster sample. There was no gypsum in the outer skim coat.

The data from the XRD analysis was processed by Rietveld Refinement, in the Maud computer program to permit quantification of the minerals present. The results obtained are presented below:

Sample Ref.	**SR2735-S1**	**SR2735-S1A**
Material	**Plaster**	**Surface Coating**
Component	**Proportion (% by Mass)**	
Calcite	96.2	98.6
Aragonite	-	1.2
Quartz	1.6	0.2
Gypsum	0.6	-
C_4AF	1.2	-
Hematite	0.4	-
Total	**100.0**	**100.0**

The sample is composed mainly of calcite (Carbonated lime), with the minor quartz, from the fine aggregate, which included a proportion of coal ash and clinker. Gypsum is present as a reaction product within the body of the plaster coat, probably between the lime in the binder and sulphates in the ash, or from the impact of environmental sulphates (soot, smoke, rising damp, etc.). The Iron Oxide is considered to be a component of the ash added to the plaster. The low hydraulic component detected is considered also to be a function of the presence of the ash in the plaster, rather than infer tah the lime was feebly hydraulic.

The thin outer surface is composed of calcite, with aragonite, which may suggest that it had received a coating of distemper or a lime wash sometime in the past.

Mix Composition

In an attempt to recover the aggregate and to ascertain the plaster composition a further sample was prepared and submitted to determination of mix composition by acid digestion. As it was indicated that some of the aggregate present may be limestone, the strength of the acid was reduced (5%) and the dwell time kept to a minimum, just sufficient to dissolve the paste. The result of the composition analysis carried out is presented below:

Sample Ref. No.	**SR 2735-S1**
Binder type (from XRD)	Non-Hydraulic Lime Lime putty
Weight proportions calculated mix ratio by dry mass.	
Lime	1.0
Aggregate	0.1
Approximate volume Proportions, calculated on the basis of the standard data for a non-Hydraulic lime Putty	
Lime	1.0
Aggregate	0.1

The aggregate from the acid digestion was recovered and the particle size distribution determined. The results of the aggregate grading are presented in the form of an aggregate filled histogram, in the appended figure No. 1 and in the table below:

Sample Reference	**SR2735 – S1 Finish Coat Plaster**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100
4.00mm	2.5	97.5
2.00mm	7.1	90.4
1.00mm	9.8	80.6
0.500mm	8.7	71.9
0.250mm	8.4	63.5
0.125mm	10.3	53.2
0.063mm	8.4	44.8
Passing	44.8	

Table No. 1: Grading analysis of recovered aggregate, following acid digestion

The aggregate particles are dominated by quartz grains, with minor limestone and clinker grains, some of which appear to contain ore minerals, along with feldspar. The particles are sub-angular and elongate to irregular in shape.

The high proportion passing the 63 micron sieve was found on examination to be a mixture of very fine quartz grains and ash/clinker components.

Microscopic Examination

To clarify the form in which the binder was used a petrographic thin section which was prepared from an intact piece of the plaster

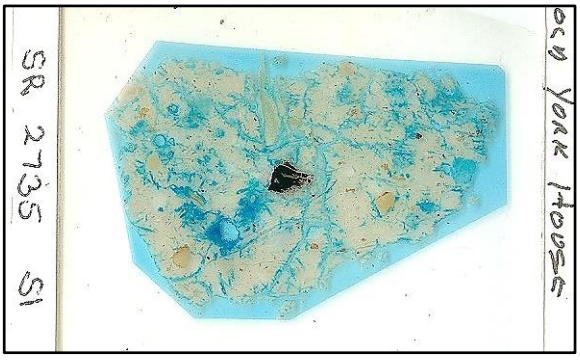

Plate No. 6:

The plate opposite shows the thin section made from the plaster for examination in the polarised light microscope.

Observations from the examination are presented below:

Aggregate

The aggregates in the plaster sample are a minor component of the mix and appear to be a mixture of very fine quartz sand (silver sand) and a proportion of ash/slag residue. In addition, there are a few limestone fragments, some which display burnt margins whilst others are fresh and unburnt.

Fragments of unburnt limestone, ranging from 0.2mm to 2.6mm were observed, and have sharp angular features, which show no evidence of burning or alteration. This would suggest that a proportion of crushed limestone was added to the mix, perhaps along with the clinker or as a seed to encourage carbonation. The limestone is mostly micritic, although small bioclastic fragments were also observed. Only one, overburnt piece of dolomitic limestone was observed in the section.

The quartz and other lithic fragments range in size from 0.05mm to 0.6mm (very fine to medium grained sand) in the section examined, although there were rare coarser fragments, up to 2.6mm in the hand specimen.

The aggregates, other than the limestone and quartz grains, are sub-angular to elongate and irregular in shape and include coal fragments, ironstone and clinker along with a proportion of coal ash. This may have been added as ash from the kiln transferred with the lime, or included in the mix as a weak pozzolan to impart a measure of hydraulicity to the plaster.

Binder

The binder is typical of that in a binder rich lime plaster, and has the appearance of a putty lime, with an abundance of shrinkage cracks distributed throughout. There are also a quantity of lime inclusions apparent throughout the plaster, with these dominated by balled putty and incompletely slaked limestone. This may infer tah the quicklime was run to a putty but not adequately screened to remove unreacted material prior to use.

The paste is fully carbonated, and the lime inclusions apparent, include both incompletely slaked lime, and rarely overburnt limestone fragments which have only partially slaked. The majority, however, are of incompletely mixed putty. The inclusions range in size from 0.22mm to 4.6mm in the section examined.

Voids and microcracks

Voids and shrinkage cracks are abundant and have the appearance of those formed within lime rich putty mixes due to entrapped air and entrained air along with an abundance of drying shrinkage cracks. The voids range in size and shape with the voids ranging from 0.06mm to 2.2mm, with these typically sub-rounds to irregular in shape. There was minimal evidence that some of the larger voids had formed to due loss of binder from the larger inclusions, although evidence of leaching was minimal.

Cracks are abundant and occur as random features distributed through the paste, peripheral to, and linking, incompletely slaked lime inclusions. The cracks are fine, ranging in width from <0.01mm to 0.14mm, and are typical of plastic shrinkage and drying shrinkage features. Although a number of them appear to be associated with bunched reinforcing fibre, which is not uncommon in plasters.

Hair

The sample contains an abundance of hair fibres with the fibres being typically 0.03 to 0.046mm in diameter and up to 38mm in length, but mostly less than 24mm. The hair is a mixture of white/grey, brown and black horse body hair, with a low proportion of reddish brown cow hair. The hair is mostly pliable and appears to be in a good condition. Based on the hair recovered from the sample used in the acid digestion, and from the modal analysis on the thin section, it was indicated the hair content of the plaster was in the region of 2.7 to 3.2kg/m³.

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

Sample Ref:	**SR2735-S1**	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	3.8	3.8
Limestone	3.6	3.6
Ash, clinker, etc	4.6	4.6
Opaque	3.0	3.0
Lime inclusions	-	9.6
Total Aggregate	**15.0**	**24.6**
Binder (Lime)	74.5	74.5
Lime inclusions	9.6	-
Secondary products/Calcite and gypsum	0.9	0.9
Total Binder	**85.0**	**75.4**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**6.0**	**6.0**
	Total	**Effective**
Binder: Aggregate Ratio	**1.0 : 0.18**	**1.0 : 0.33**

Table No. 2: Result of modal analysis (900-point count) on the thin section

Photomicrographs:

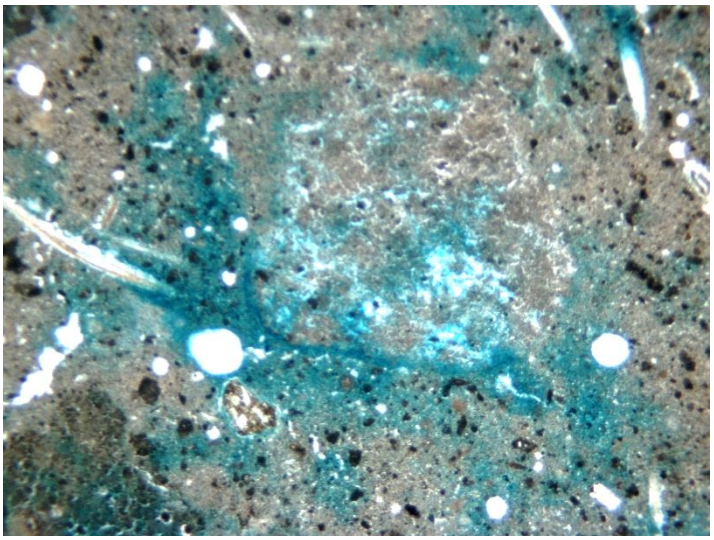

Plate No. 7:

A view in plane polarised light (ppl) showing a typical area of the plaster, with a fully calcined and slaked putty lime inclusion on the centre of the plate.

The paste is fully carbonated and contains mostly quartz fine aggregate particles, and a limestone fragment, in the lower left corner.

Hair fibre can be seen in the centre left and in the upper right corner.

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

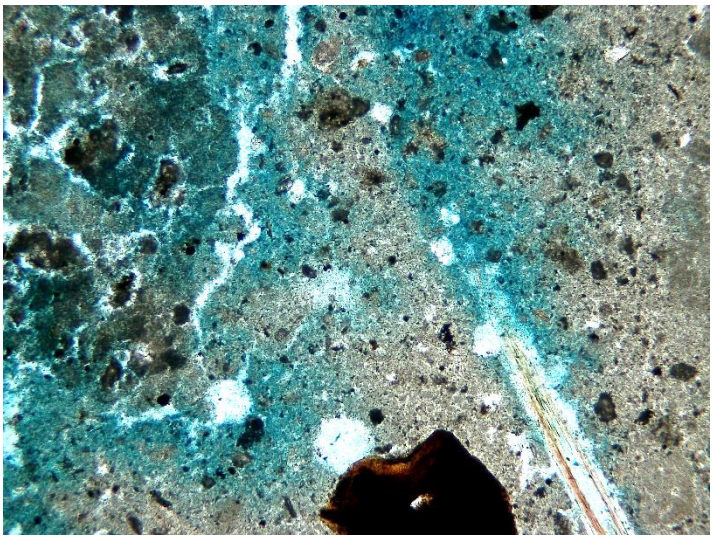

Plate No. 8:

Another view of sub-angular inclusions within the paste, on the left side. This view is again in ppl. The paste adjacent to the inclusion is particularly microporous and contains shrinkage cracks.

The aggregates are dominated by very fine quartz particles along with an opaque particle, ironstone (black in plate) in the margin. Fine ash and slag particles (brown) can be seen distributed throughout. A hair fibre is in the lower right side.

Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

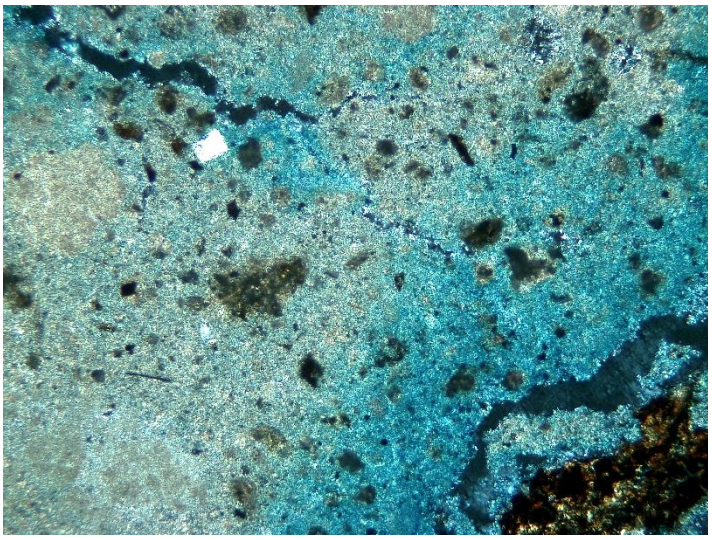

Plate No. 9:

A view in Cross polarised light xpl, of an area containing a lime inclusion that appear to be partially overburnt, and partially hydrated, lower right corner. The paste is fully carbonated and cracks within the inclusion are lightly fringed with redeposited calcite.

A number of small pozzolanic/clinker particles can be seen with some displaying reaction rims, see left of centre and upper right.

Porosity, voids and impregnating resin all appear dark in xpl. Field of view 1.2mm.

Summary

From the examination and analysis of the plaster sample from the internal walls of the Old York House, Malton. North Yorkshire, it is indicated that the mortar was made from a non-hydraulic air lime and a mixture of fine natural sand (silver sand) along with a proportion of ash/slag fines, which were added either accidentally, with the binder, or intentionally as a pozzolan. The lime having been used in the form of an unscreened putty contained a small proportion of unburnt and partially slaked limestone particles. Details of the mix used is summarized below:

Sample Ref.	**SR2735- S1**
Volume Proportion by acid digestion	
Binder content (Putty)	1.0
Fine Aggregate (Sand+Ash/clinker)	0.1
By modal analysis	
Binder: Agg. by vol. (Total)	1.0 : 0.18
(Effective)	1.0 : 0.33
Hair Content	2.7 to 3.2kg/m³

The aggregates in the plaster is a mixture of a natural fine quartz sand, with a proportion of ash/slag/clinker.

Sample Reference	**SR2735 – S1 Finish Coat Plaster**	
British Standard Sieve Size	**Percentage Retained**	**Percentage Passing**
8.00mm	0	100
4.00mm	2.5	97.5
2.00mm	7.1	90.4
1.00mm	9.8	80.6
0.500mm	8.7	71.9
0.250mm	8.4	63.5
0.125mm	10.3	53.2
0.063mm	8.4	44.8
Passing	44.8	

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 received in CMC Ltd.'s laboratory on the 19th June 2019, from Nigel Copsey of the Earth, Stone and lime Company, which was identified as having been sampled from the finish coat plaster in the Old York House, Malton, North Yorkshire.

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For CMC Ltd.

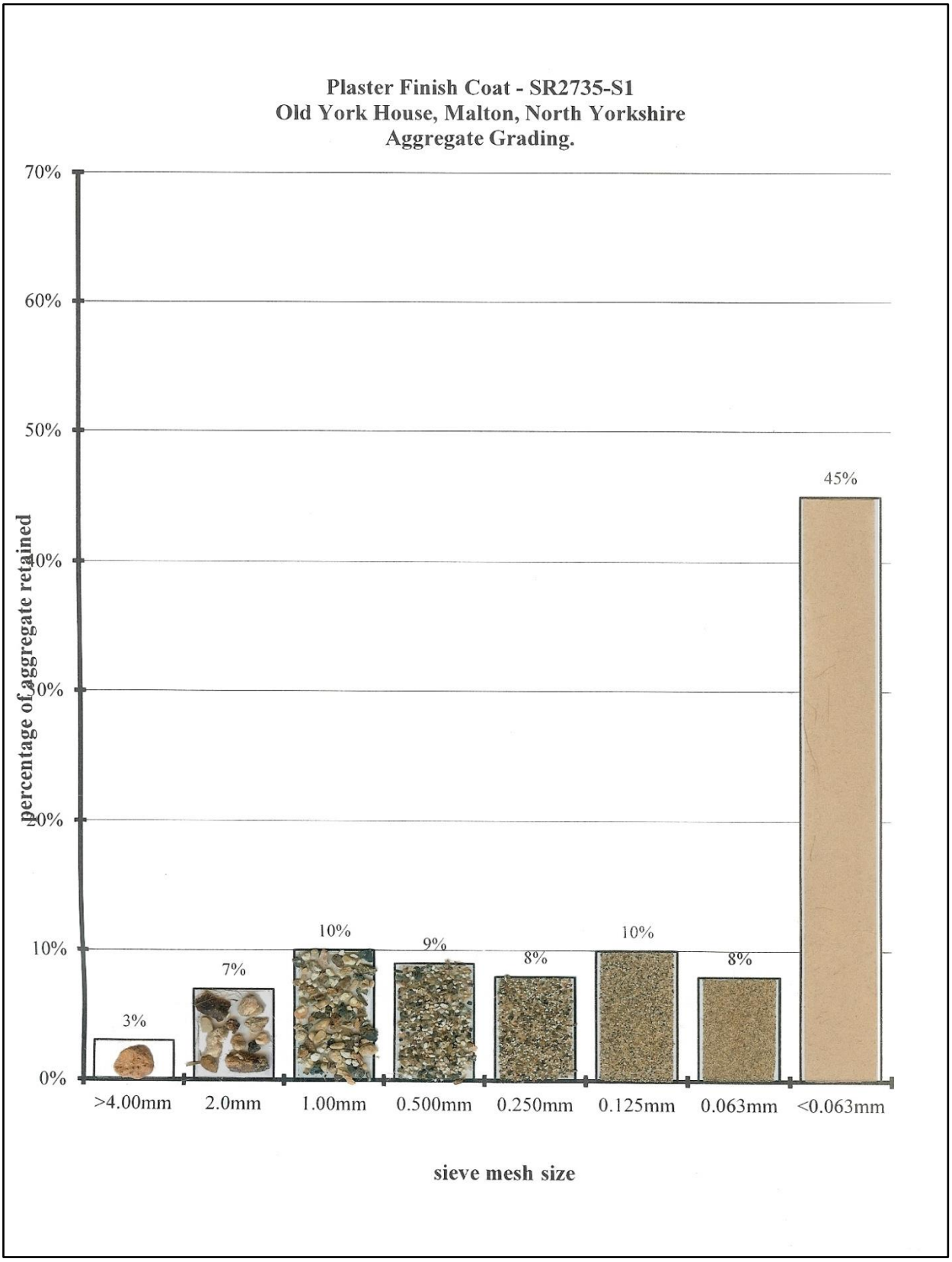

 Figure No. 1 – Grading of recovered aggregate - Sample SR2735-S1

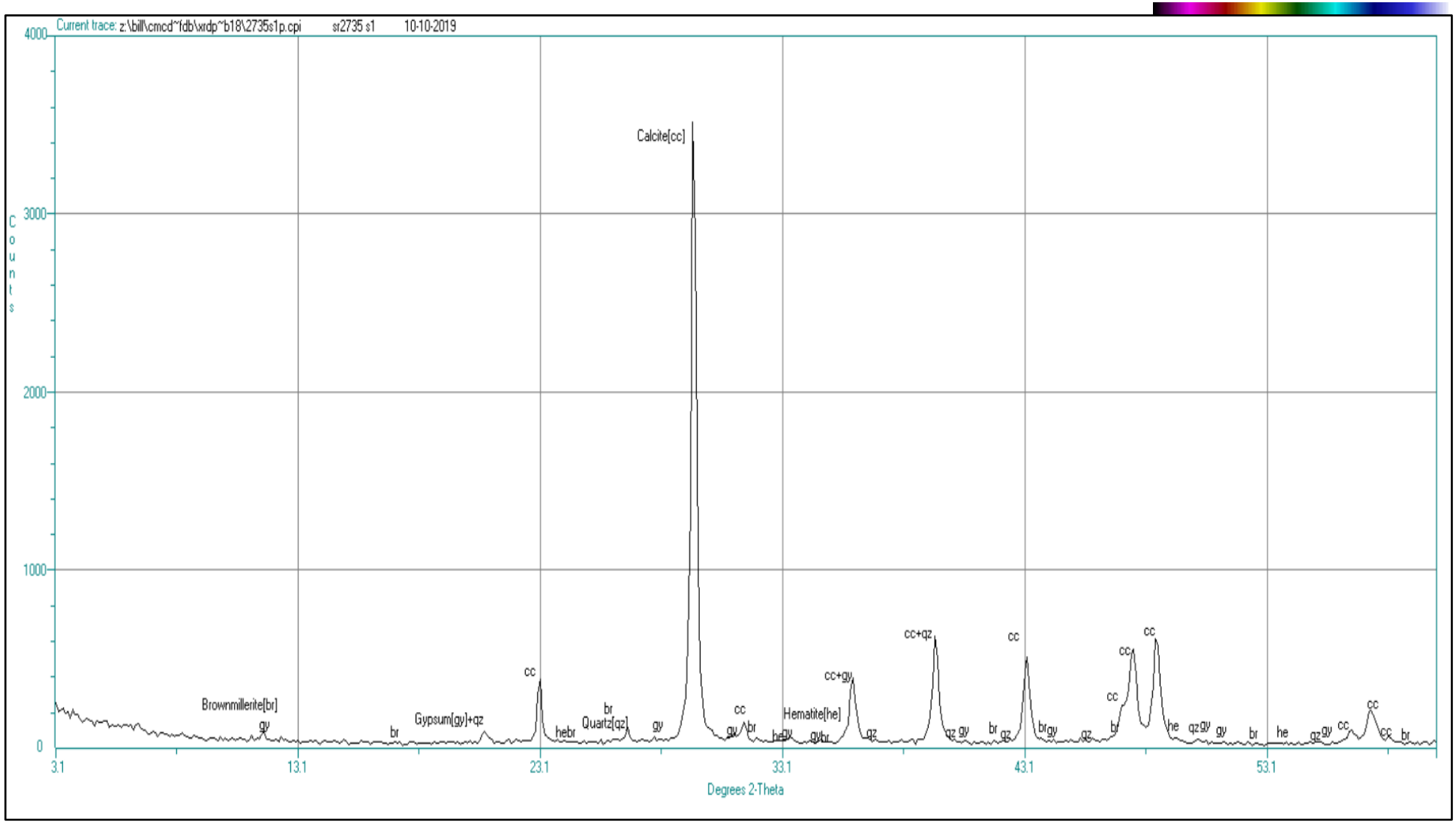

Figure No. 2: Sample SR2735-S1 – Internal Finish Coat Plaster, ex The Old York House, Malton.

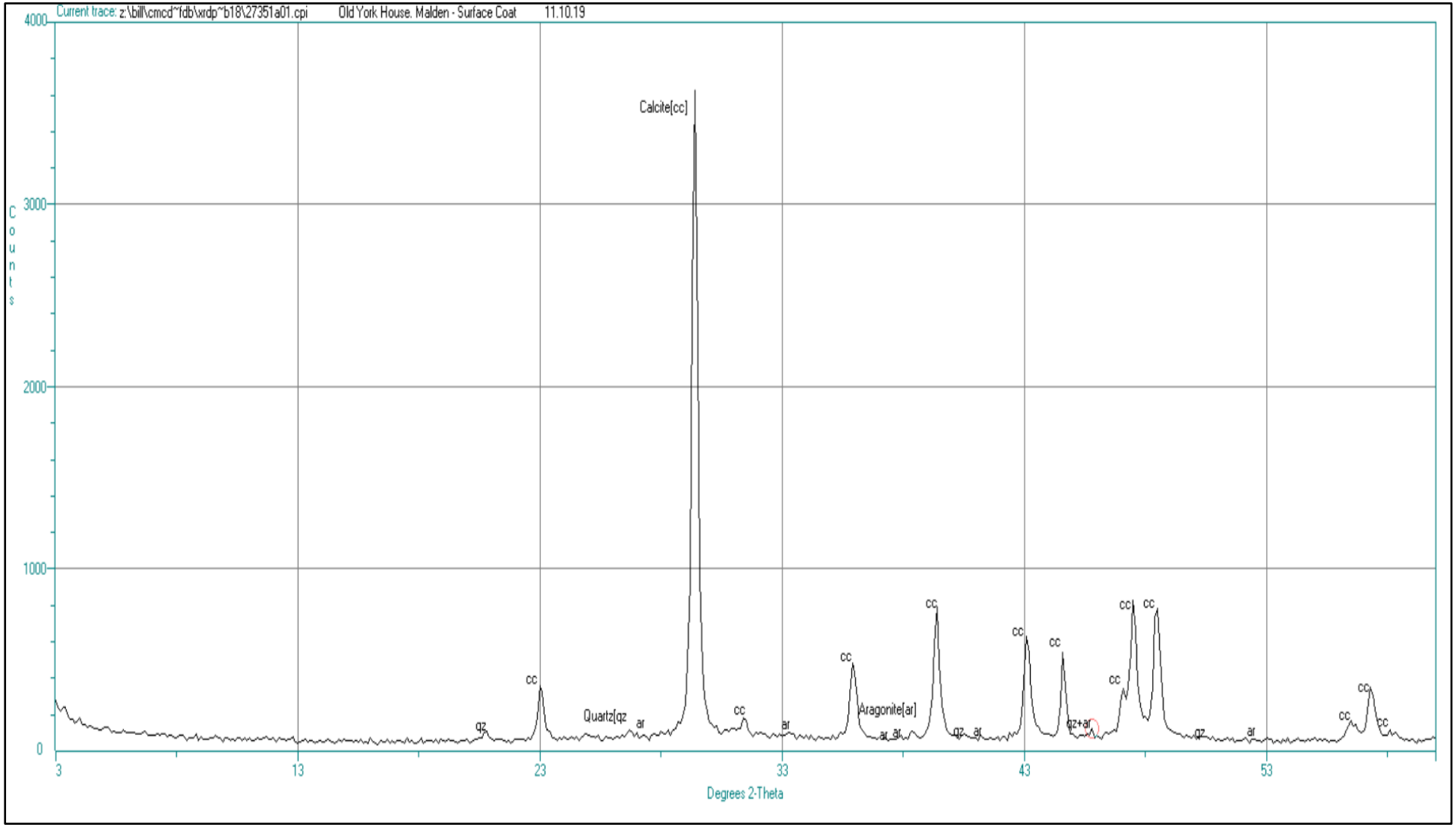

Figure No. 3: Sample SR2735-S1a – Thin coating on outer surface of Finish Coat Plaster.