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Our Ref: M/2106/21/C1
Your Ref.: Old Sun Inn

4th July 2021

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

Project Reference	:	Old Sun Inn, Spout House Farm, Bilsdale North York Moors National Park.
Sample Source	:	16th Century Thatched Cruck-framed House
Sample Description	:	Pointing mortar over Earth-lime mortar to Gable
Date Received	:	8th April 2021
CMC Sample Ref	:	SR 2833 - S1
Method of Test	:	Mix composition fabric condition assessed from a Thin Section examination, with binder type by XRD analysis following in-house procedures.

Sample

A sample of mortar from Nigel Copsey of the Earth, Stone & Lime Company, was received. in CMC's Stirling laboratory on the 8th April 2021, with the sample identified as pointing mortar applied over a clay/lime building mortar, and taken from the 16th *c* gable of the Old Sun Inn, Spout House Farm, in the North York Moors National Park.

Information relating to the sample was received by email on the 14th February 2021, with confirmation that the sample was despatched to CMC on the 6th April 2021, along with the instruction that it was to be submitted to analysis to establish the composition of the mortar, its condition, and any other useful information that may be gleaned from the examination and analysis.

On receipt in the laboratory, the sample details were entered into the sample register and the unique sample identification number SR2833 allocated.

The details of the sample received is presented below:

CMC Ref.	Client Ref	Location Sampled
SR2833 - S1	Pointing Mortar	Pointing mortar from the 16th *C* Gable of the Old Sun Inn, Spouts House Farm, Bilsdale, North York Moors National Park.

Method of Test

Prior to preparing the sample for analysis the sample was photographed on receipt, with its mass and size recorded. The sample was then submitted to an examination with the aid of a stereo-binocular microscope at a magnification up to x10, during which the sample was exposed to a series of *ad hoc* droplet tests employing a range of reagents and indicator solutions. This was to aid the identification of the components present and to assess the condition of the mortar sample as received.

To permit confirmation that the binder used in the production of the mortar was a non-hydraulic lime, as intimated, a representative sub-sample was prepared for analysis by X-ray Diffraction (XRD). The sub-sample consisted of several pieces from each of the intact mortar lumps, which were gently ground in an agate mortar and pestle to separate the binder from any aggregates that may be present. A binder rich sub-sample was then obtained by sieving the disaggregated material over a 63µm sieve.

The powder passing the sieve was backpacked into a proprietary sample holder in preparation for presentation in the diffractometer. With the sample 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. The digital output from the diffractometer was 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 slice was cut from the largest intact piece of mortar and used for the preparation of a petrographic thin section. This was required as it was indicated from the visual examination that the mortar contained limestone aggregate, and as the mix proportion could not be determined by acid digestion, the thin section was, therefore, used to assist in establishing the form in which the binder was used, in the production of the mortar, and to permit determination of the mix composition by modal analysis.

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
SR2833-S1	Mortar	73.4	50.1 x 29.4 x 20.2	4 + fines	"White"

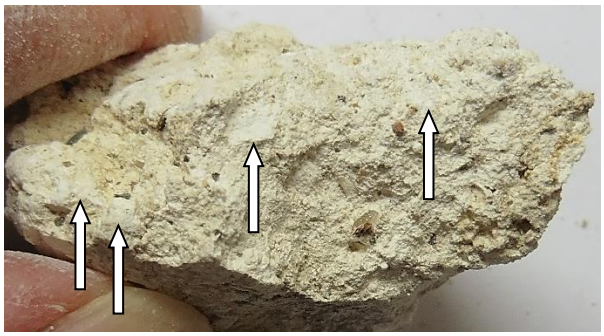
The sample received consisted of four intact pieces of mortar along with a small quantity of fines. The mortar pieces were soft to moderately hard and well compacted, only requiring firm finger pressure to break, though once disrupted the mortar could be powdered further under moderate to light finger pressure.

It was observed that all of the intact pieces contained an abundance of limestone fragments bound in a lime paste, in which several lime inclusions also appeared to be present. All four fragments were visually very similar. The inclusions were noted to range from sub-angular to sub-round and irregular in shape. The largest inclusion was measured at 4.2mm, but most were finer than 1.5mm in size.

On testing a freshly fractured surface, with a phenolphthalein indicator solution, the mortar, and the inclusions, were found to be fully carbonated, with no colour change of the indicator observed.

Water droplet tests were carried out, which involved placing droplets from a pipette onto both the outer mortar surfaces and onto freshly fractured surfaces. The test confirmed that the mortar pieces all contained a well-connected pore structure, with the droplets, placed absorbed very quickly and diffused through the fabric of the mortar to depth.

Plates No. 1 & 2: The left plate above shows the four intact pieces in the mortar sample as received. The right plate shows the intact piece selected for thin section preparation.



Plates No. 3 & 4: The above plates show freshly fractured surfaces through two pieces of mortar. The left plate is a close-up of one fragment showing the abundance of fine limestone fragments bound in a lime paste with an angular lime inclusion, arrowed in plate. The right plate shows another view of a freshly broken surface through the thickness of another fragment. Again, note the presence of white soft lime inclusions, arrowed in the right plate.

In addition, to the limestone aggregate there was a low abundance of other natural aggregates observed, these included quartz grains and opaque particles, the latter possibly coal or ironstone.

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 collected and prepared for analysis by X-ray Diffraction (XRD). This would assist in confirming if any hydraulic components were present in the lime, and also identify if the were any mineral additions (pozzolans), or reaction products present, i.e., sulphates.

The result of the XRD analysis is presented in the following figure, in the form of a labelled X-ray Diffractogram at the end of this certificate:

Figure No. 1: Sample SR2833-S1 – Binder rich sub-sample from Pointing mortar, ex the Old Sun Inn.

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

cc = Calcite ($CaCO_3$) calcium carbonate, carbonated lime binder, and also the dominant component of the limestone present in the aggregate,

qz = Quartz (SiO_2) dominant aggregate component on the fine sand grains, with possibly some present as trace proportion within the limestone.

On the basis of the XRD analysis, it is indicated that mortar sample was made from an air lime binder, with no evidence of any hydraulic components present. Trace proportions of feldspars of the Plagioclase Group of minerals were detected, along with Dickite, a clay mineral of the Kaolinite group, but these were of insufficient quantity to infer that they were present as components of the mix and were most likely present as residue from surface, or substrate, contaminants.

The mortar is fully carbonated and there was no evidence of the mortar having been affected by reactions with any form of environmental sulphate, or other deleterious contaminants.

To provide additional information the data from the XRD analysis was processed by Rietveld Refinement, in the Maud computer program, to permitted quantification of the minerals and crystalline material present. The results obtained are presented below:

Sample Ref.	**SR2823-S1**
Material	**Lime Inclusion**
Component	**Proportion (% by Mass)**
Calcite ($CaCO_3$)	95.6
Quartz (SiO_2)	4.3
Feldspar (Anorthite)	<0.1
Dickite (Clay)	<0.1
Total	**100.0**

The mortar is composed of calcite (Carbonated lime and limestone), along with a minor proportion of quartz, and trace proportions of feldspar and clay, although the latter two are not identified in figure No. 1, due to their low concentration.

Microscopic Examination

To clarify the form in which the binder was used and permit comment on the mortar fabric a petrographic thin section was prepared from an intact piece of the mortar and submitted to a microscopic examination.

The petrographic thin section was submitted to a microscopic examination in an Olympus BH2 polarised light microscope, which was fitted with a digital camera to permit recording of features of interest, a selection of which are included in this report for reference. The examination was carried out in general accordance with the methods detailed in the "Petrographic Examination of Mortars, Plasters, Renders and Related Materials – Applied Petrography Group of the Geological Society of London", published February 2008.

Plate No. 5:

Thin section prepared from a representative piece of the pointing mortar to allow for its examination in the petrographic microscope.

Aggregate

The aggregates in this mortar sample are dominated by limestone fragments with a low proportion of other lithic particles. The latter are dominated by quartz grains, along with minor quartzite, fine grained sandstone and siltstone and rare altered indeterminate lithic fragments. There are also trace proportions of opaque minerals, ash clinker and pieces of wood. It is not possible to clearly identify all of the opaque minerals by optical microscopy alone, although ironstone and coal fragments are both indicated to be present.

The limestone fragments are typically angular to sub-angular, with the quartz and other lithic fragments typically sub angular to irregular in shape. The latter displaying rounded, water worn, margins.

The limestone fragments have the appearance of having been crushed with most of the larger angular fragments containing Ooliths and bioclasts, and it is considered that they were added to the mortar in the form of crushed Oolithic limestone fines. However, a very small proportion of limestone fragments displayed dolomitic textures, though their low abundance would suggest that they may have originated as a contaminant rather than infer that the source was a dolomitic limestone, though it may contain a thin seam of dolomitic material.

The quartz and lithic sand grains range from 0.06mm to 0.8mm in size, whereas the limestone fragments range in size from 0.05mm to 1.5mm in size, and most show sharp fractured margins.

Binder

The paste has the appearance of having been produced from a non-hydraulic lime, with a proportion of sub-angular to sub-round lime inclusions, along with a proportion of partially burnt and overburnt particles also found to be present.

Lime inclusions are variably slaked, with rare fragments observed to retain residual rock fabric, which would infer that calcining of the limestone was, in part, incomplete, along with the shape and texture of others inferring that they had been locally poorly slaked at the time of mixing.

The variation in shape, of the inclusions, along with the well compacted encapsulating paste inferred that the mortar had most likely been mixed as a Hot Mixed Mortar (HMM), where the quicklime was mixed with the crushed limestone, but mixed with sufficient water to almost fully slake the lime to a putty.

However, the retention of incompletely slaked lime along with a paste having the texture typical of a putty lime, would suggest that the mix was perhaps allowed to allowed to cool, and remain as a wet mix, prior to being remixed before placing. During remixing some of the unhydrated quicklime may then have been reactivated, thereby imparting the localised hot placed mortar (HPM) characteristics observed. The paste is fully carbonated throughout the sample and there is evidence of localised leaching, with loss of lime both from inclusions and locally from the paste along crack paths which have acted as water percolation pathways. However, only little, very localised, redeposition of calcite, was observed, with this along crack margins and lining voids from which inclusion had been depleted.

The lime inclusions observed ranged in size from 0.8mm to 3.9mm in the section examined, but mostly these are <2.0mm.

Voids and microcracks

The voids observed in the mortar are mostly sub-angular to irregular in shape, and are a mixture of dissolution voids from the post placement depletion of lime inclusions, and entrapped air voids. The voids range in size from 0.2mm to 1.3mm in size, a few of which retain Calcite rims from leached and redeposited lime.

Cracks are abundant, with those present having the appearance of early drying shrinkage cracks, typical of those found in air lime mortars. Cracks range in width from <0.01mm to 0.05mm.

Modal Analysis

The mix proportion from the modal analysis are reported as both Effective and Total binder content to aggregate ratio, by volume. See the following table for proportions:

Sample Ref:	**SR2833-S1**	
Constituents	**%**	
Aggregate	**Inclusions as binder**	**Inclusions as Aggregate**
Quartz	4.1	4.1
Lithic	1.7	1.7
Sandstone/Siltstone	1.5	1.5
Limestone	22.1	22.1
Dolomitic Limestone	0.6	0.6
Ash & clinker	2.4	2.4
Lime inclusions	-	5.7
Total Aggregate	**35.5**	**41.2**
Binder (Lime)	58.2	58.2
Lime inclusions	5.7	-
Secondary products/Calcite	0.6	0.6
Total Binder	**64.5**	**58.8**
Total Constituents	**100.0**	**100.0**
Cracks/Voids	**12.0**	**12.0**
	Total	**Effective**
Binder: Aggregate Ratio	**1.0 : 0.55**	**1.0 : 0.70**

Table No. 1: Result of modal analysis (600-point count) on the thin section.

Photomicrographs:

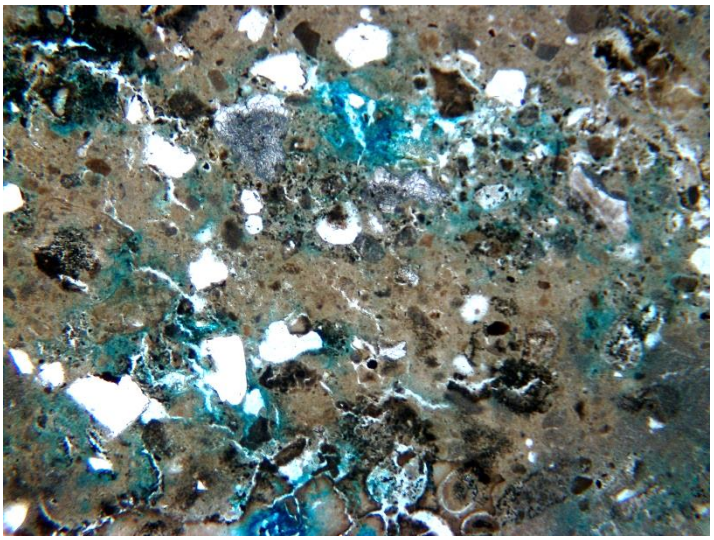


Plate No. 6:

A view in plane polarised light (ppl) of a quartz aggregate rich area of the mortar fabric. The paste containing an abundance of shrinkage cracks typical of an air lime mortar placed in a putty consistency. The paste is fully carbonated and encapsulates a mixed suite of aggregates, dominated by quartz particles, white in plate, along with siltstone, coal/ironstone and ash fragments

Porosity and voids are highlighted by the blue dyed resin.

Field of view 2.4mm.

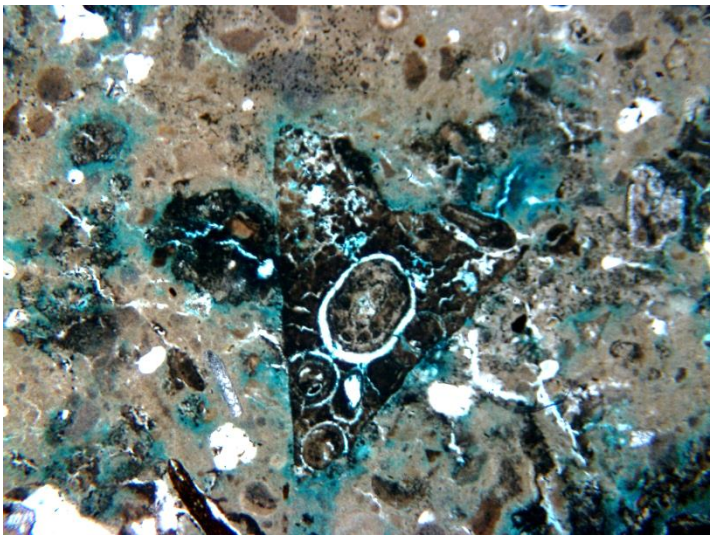


Plate No. 7:

Another view in plane polarised light (ppl) where an angular limestone fragment can be seen in the centre part of the plate. The limestone is from a bioclastic rock containing Ooliths, shell fragments and foraminifera, etc. The aggregates in view on are dominated by quartz grains, with opaque minerals and ash clinker.

Porosity and voids are highlighted by the blue dyed resin.

Field of view 2.4mm.

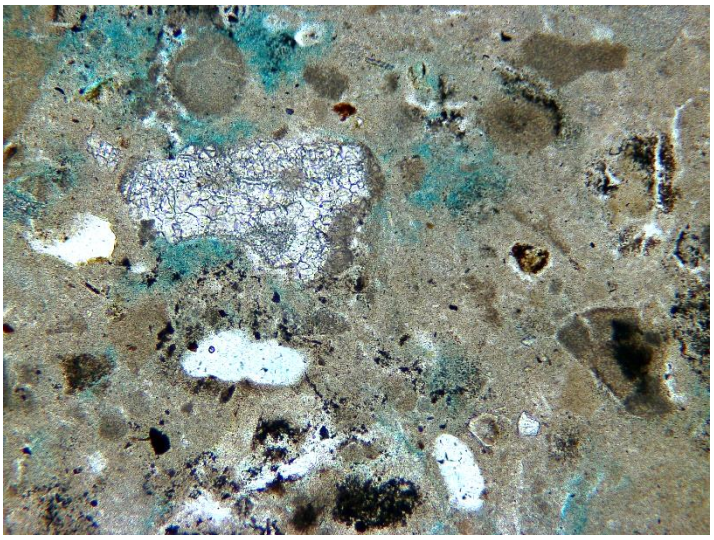


Plate No. 8:

Another view in plane polarised light, ppl, where a small dolomitic limestone fragment can be seen in the centre left of the plate. The surrounding paste is relatively compact and is fully carbonated with a moderately high microporosity. The aggregates in view are again dominated by quartz grains, mostly white in this image, with minor opaque minerals. Small limestone dust concentrations can be seen distributed throughout.
Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

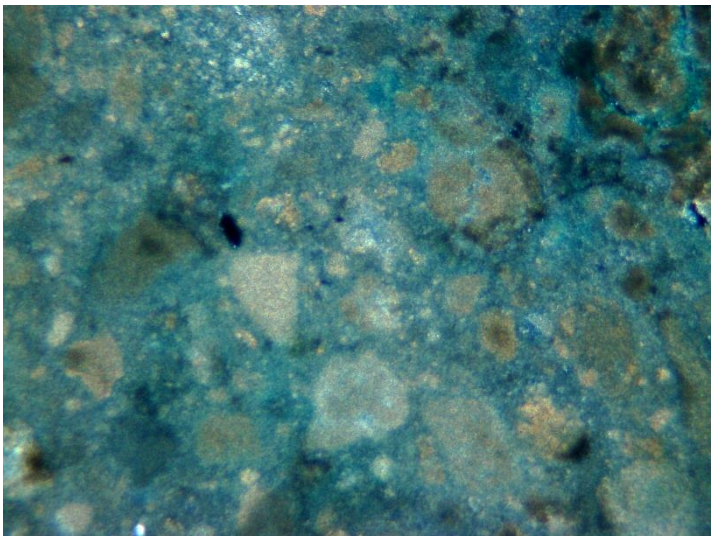


Plate No. 9:

A view in cross polarised light, xpl, showing an area of the fabric containing an abundance of fine limestone fragments distributed throughout the lime paste. The paste displays a high microporosity. Ash fragments can be seen in the upper right corner. No natural aggregates are present in this view

Porosity, voids, impregnating resin and opaque minerals all show dark in xpl.

Field of view 1.2mm.

Summary

From the examination and analysis of the mortar sample from the gable of the old Sun Inn, it is indicated that the mortar was made from a non-hydraulic air lime and crushed limestone fines. The lime having been used in the mortar production in the form of a quicklime. The mortar shows all the features of having been mixed as a Hot Mixed Mortar, although it appears to have been allowed to cooling, and remain as a wet mix, i.e., not slaked to dryness. With the mortar remixed, with some latent slaking occurring, as it was placed.

Details of the mix used is summarized below:

Sample Ref.	**SR2833- S1**
Volume Proportion by modal analysis	
Binder: Agg. by vol. (Total)	1.0 : 0.55
(Effective)	1.0 : 1.6

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 Ltd.'s laboratory on the 8th April 2021, from Nigel Copsey of the Earth, Stone and Lime Company, which was identified as Pointing mortar from the Old Sun Inn, Spout House Farm, North York Moors National Park.

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For CMC Ltd.

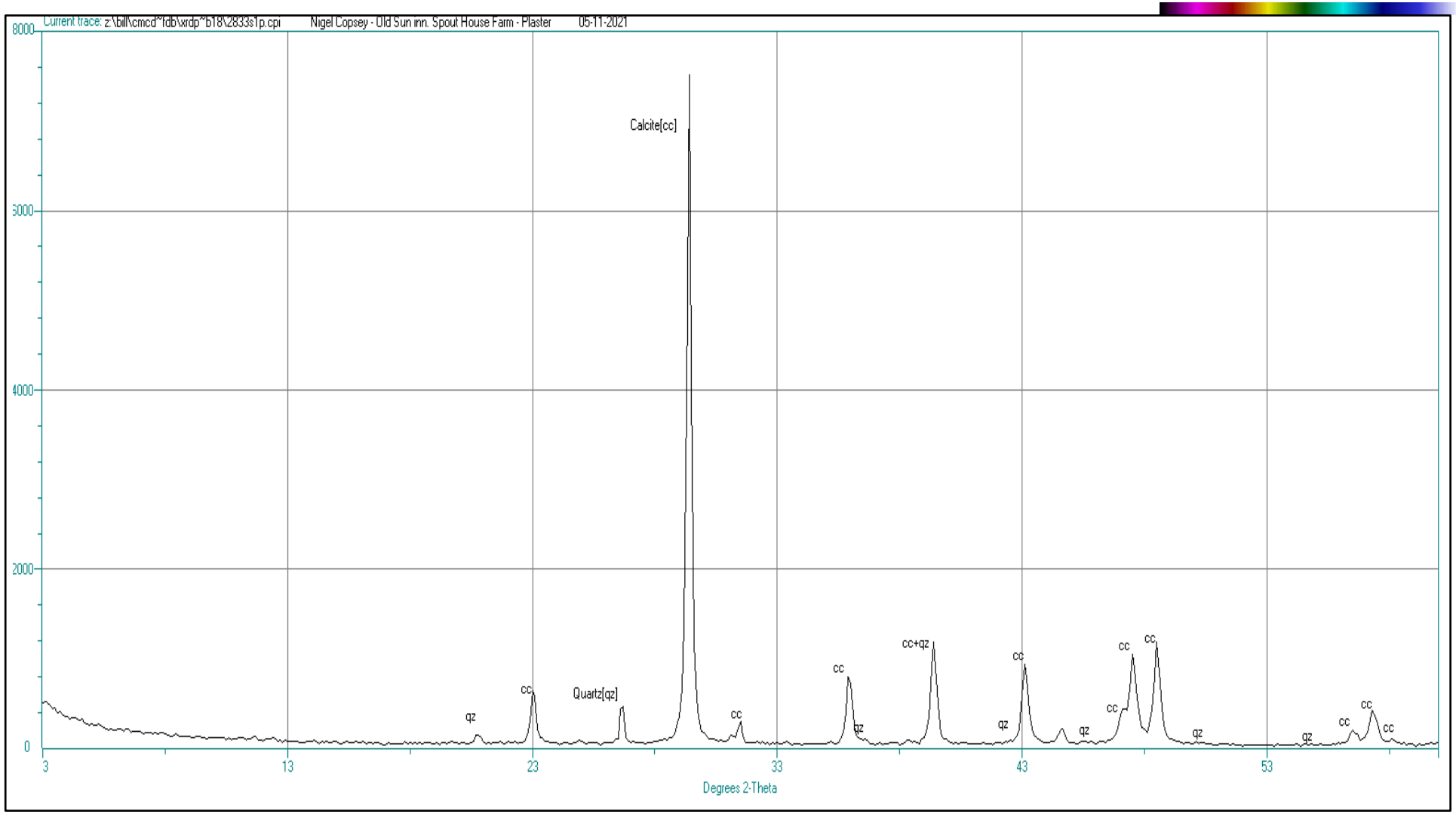

Figure No. 1: Sample SR2833-S1 – Binder rich sub-sample from Pointing mortar, ex the Old Sun Inn.