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The Earth, Stone & Lime Company.
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Our Ref: M/2107/21/C1
Your Ref.: Thornton Lime

4th July 2021

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

Project Reference	:	Thornton Lime Mortar
Sample Source	:	Hall Farm, Maltongate
Sample Description	:	11-month-old Plaster mixed from lime produced by burning local Oolitic Limestone
Date Received	:	8th April 2021
CMC Sample Ref	:	SR 2834 - S1
Method of Test	:	Binder type by XRD analysis by in-house procedures, with fabric condition and mix composition from thin section examination.

Samples

A sample of plaster was received in CMC's Stirling laboratory on the 8th April 2021. The sample was identified as plaster from a trial mix prepared by Nigel Copsey of the of the Earth, Stone & Lime Company.

The sample was provided for the purpose of gaining further knowledge as to the performance of the quicklime for use in plaster and masonry mortar.

On receipt in the laboratory, the sample details were entered into the sample register and the unique sample identification number SR2834 allocated, see below for details:

CMC Ref.	Client Ref	Material / Location Sampled
SR2834 - S1	Plaster	Plaster made from quicklime prepared by calcining local Oolitic Limestone

Method of Test

Prior to preparing the sample for analysis it was photographed on receipt in the laboratory, 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 this 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 sample as received.

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

The powdered sample 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, an intact piece from the sample was used in the preparation of a petrographic thin section. This to assist in establishing the condition of the binder and the distribution of the hair throughout the paste.

Observations from Macro/microscopic examination

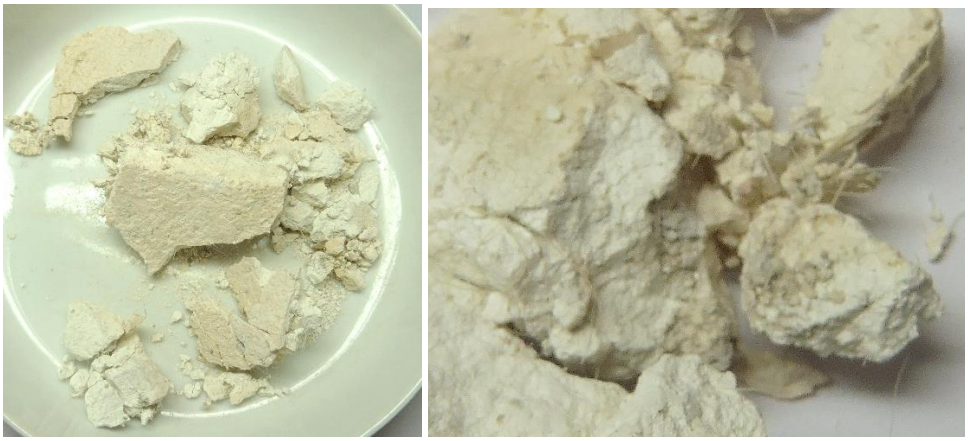
The sample was logged on receipt with the following determined:

Sample Ref.	Client Ref.	Mass of Sample (gram)	Dimensions of Largest piece (mm)	No. of Pieces	Colour by the Munsell Colour Charts
SR2834-S1	Plaster	75.6	53.0 x 36.6 x 25.0	11 No	"White"

The sample consisted of 11 pieces of plaster. The intact pieces were well compacted pieces of hair reinforced plaster. The plaster was soft to firm to the touch though it was easily broken and powdered under light to moderate finger pressure. This is not unexpected as the plaster had only been aged for 11 months prior to submitting it for analysis.

On testing a freshly fractured and sawn surfaces with a phenolphthalein indicator solution the plaster, was found to be only partially carbonated, with a patchy colour change observed.

Water droplet tests, which involved placing droplets from a pipette onto outer plaster surfaces and freshly fractured surfaces, confirmed that the plaster contained a well-connected pore structure, with the droplets absorbed very quickly and diffused through the plaster to depth on all surfaces tested.



Plates No. 1 & 2: The above plates show the sample as received, left plate, and a close-up of a fractured surface showing the presence of hair, right plate.

Plates No. 3 & 4: The above plates show two of the fractured edges of the sample, with the left plate showing the fibre added to the plaster, which has the appearance of a synthetic fibre. The right plate shows the result of the phenolphthalein indicator test, the colour change indicating incomplete carbonation.

There were no aggregates within the body of the plaster and it was reported that it was made from slaked quicklime and hair.

Results of XRD Analysis

To confirm if the binder was a lime, and determine if there were any hydraulic components present, a binder rich sub-sample of the paste was obtained and submitted to analysis by X-ray Diffraction (XRD).

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

Figure No. 1: Sample SR2834-S1 – Binder from plaster made at Hall Farm, Maltongate.

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

- **po** = Portlandite ($Ca(OH)_2$) calcium hydroxide, form of lime after slaking and before carbonation,
- **cc** = Calcite ($CaCO_3$) calcium carbonate, carbonated binder from lime plaster, and also any limestone present as aggregate,
- **qz** = Quartz (SiO_2) component of any the aggregate, can also be present within some limestones,
- **fs** = Feldspar, mostly of Anorthite of the Plagioclase group of minerals, trace proportions only, probably present as a contaminant in the limestone.
- **di** = Dickite, clay mineral of the Kaolinite group, present in trace proportions, within limestone,
- **ett** = Ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}26H_2O$) Calcium Aluminium Sulphate Hydroxide Hydrate, present as a reaction product from the impact of environmental sulphates, or a contaminant.

On the basis of the XRD analysis, it is confirmed that the plaster sample was made from a non-hydraulic, high calcium, air lime. The plaster is only partially carbonated, which given its age, is expected. However, the presence of Ettringite was unexpected, and would only be expected had there been a gauging of a hydraulic component or the plaster had been in contact with a cementitious material and/or been affected by environmental sulphates. Alternatively, the components necessary for its formation may be present from the burning process, with these introduced into the quicklime from the burning of coal, if the fuel used to fire the kiln was indeed coal.

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.	**SR2831-S1**
Component	**Proportion (% by Mass)**
Portlandite	72.1
Calcite	26.5
Quartz	0.4
Feldspar (Anorthite)	<0.1
Dickite	<0.1
Ettringite	0.8
Total	**100.0**

The binder is composed of Portlandite (Calcium hydroxide, and its carbonated form, calcite (Carbonated lime), and it is indicated that the lime used in the production of the mortar was a high calcium lime with no hydraulic components detected.

The quartz and feldspar detected are present as contaminants, or from the limestone used in producing the quicklime.

It is considered that the Ettringite was formed due to contamination of the quicklime during the firing of the limestone in a coal fuelled kiln.

Microscopic Examination

To provide information on the form and condition of the binder and permit comment on the plaster fabric a petrographic thin section was prepared from an intact piece of the sample.

Plate No. 5:

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

Although it was reported that there was no aggregate in the sample, two small clusters of very fine quartz grains along with plagioclase feldspar were observed. These, however, were apparently encapsulated within two separate fragments of calcined limestone. Inferring that the limestone source contained horizons of ‘dirty’ limestone.

Three unburnt limestone fragments were also observed, which are all from an Oolitic limestone, two of the fragments shows no evidence of the burning of their outer surfaces, whilst the other is partially burnt along one third of its length, i.e., 0.8mm.

Binder

The binder has the appearance of a non-hydraulic lime, with a proportion of incompletely calcined and/or incompletely slaked lime/limestone inclusions observed within the plaster fabric. The inclusions range from sub-angular to irregular and sub-rounded and most show partial diffusion into the surrounding paste. However, a minor proportion of the inclusions display sharp outer margins, and do not diffuse into the encapsulating paste around at least part of their outer margin.

The variation in shape of the inclusions, and the appearance of the paste, would infer that the quicklime had been slaked to a putty, but perhaps not fully screened to remove all of the partially burnt or incompletely slaked limestone particles.

The paste is only partially carbonated with carbonation observed around the margins of the piece used in the thin section preparation. With locally invasive carbonation into the plaster along fine crack paths and via microporous areas formed around the margins of inclusion.

The lime inclusions observed ranged in size from 0.4mm to 3.6mm in the section examined, and there are several inclusions present that retain a clear rock imprint along with an abundance of separated Ooliths and, therefore, it can be confirmed that the limestone used in the lime production was an Oolitic limestone.

Voids and microcracks

The voids are sparse with those observed in the plaster being sub-round to irregular in shape, and appear to be entrapped air voids. The voids range in size from 0.04mm to 1.2mm in size. A proportion of the smaller voids retain very fine Calcite margins, this deposited from the evaporation/absorption of mixing waters, rom the depletion of excess slaking water.

Cracks are minor, with those present having the appearance of early drying shrinkage cracks. With those within paste rich areas being typical of the form commonly associated with lime rich putty lime plasters. Cracks range in width from <0.01mm to 0.02mm, and are typical early shrinkage cracks.

Fibre Reinforcement

The fibre was not extracted and submitted to separate microscopic examination, though it was observed to be well distributed throughout the plaster, with localised clustering apparent, this is typical in all haired plasters. However, unlike most traditional plasters the sample received did not contain any clumps, inferring that hair was well separated and added to the plaster in such a way as to be evenly distributed throughout the mix.

Photomicrographs:

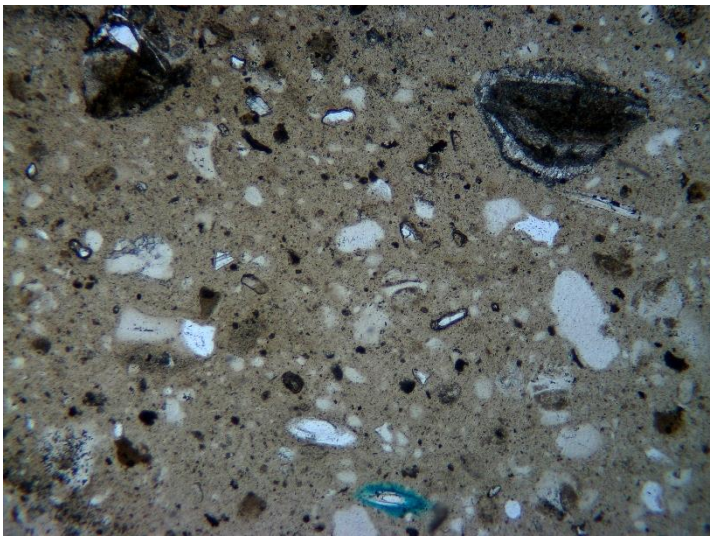


Plate No. 6:

A view in plane polarised light (ppl) of a typical area within the plaster. The paste is relatively dense and is partially carbonated in this view. There is a quantity of very fine limestone dust fragments distributed throughout, brown in image, along with two larger limestone fragments, upper left and right . The voids are a mixture of small entrapped air voids with some also the imprint of the hair fibre included in the paster. Where cut perpendicular to the plane of the slide, they appear as round to elliptical holes. Porosity and voids are highlighted by the blue dyed resin. Field of view 2.4mm.

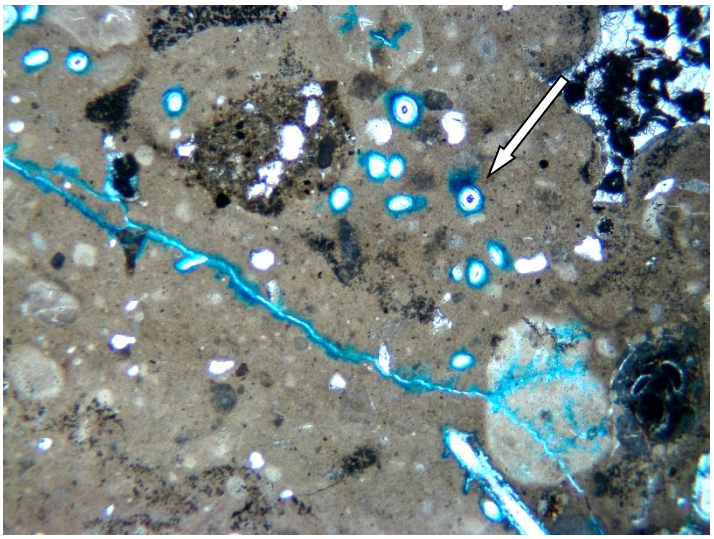


Plate No. 7:

Another view in ppl, where a limestone inclusion can be seen in the upper right. This inclusion appears not to have been calcined, whereas that in the lower right is fully calcined and slaked inclusion is present as a porous putty inclusion. A crack transects the plate, with no evidence of moisture percolation through it. A cluster of hair fibres can be seen in the upper centre, one example is arrowed in the plate. An overburnt particle can be seen lower right with an ash cluster in the upper left.
Porosity and voids are highlighted by the blue dyed resin. Field of view 2.4mm.

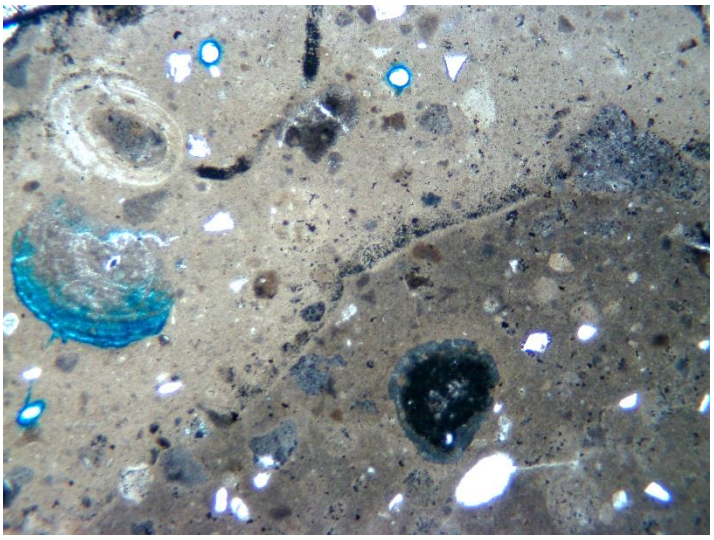


Plate No. 8:

A view in plane polarised light, ppl, of the interface between carbonated paste, light brownish grey, and uncarbonated lime, dark greenish grey, lower right side of plate. These are separated by a distinct boundary which would infer that the uncarbonated paste is due to the denser fabric, formed by an incompletely slaked dense lime inclusion. Note the presence of two Ooliths, upper left, which are encapsulated within the fully slaked and carbonated encapsulating paste.
Porosity and voids are highlighted by the blue dyed resin. Field of view 1.2mm.

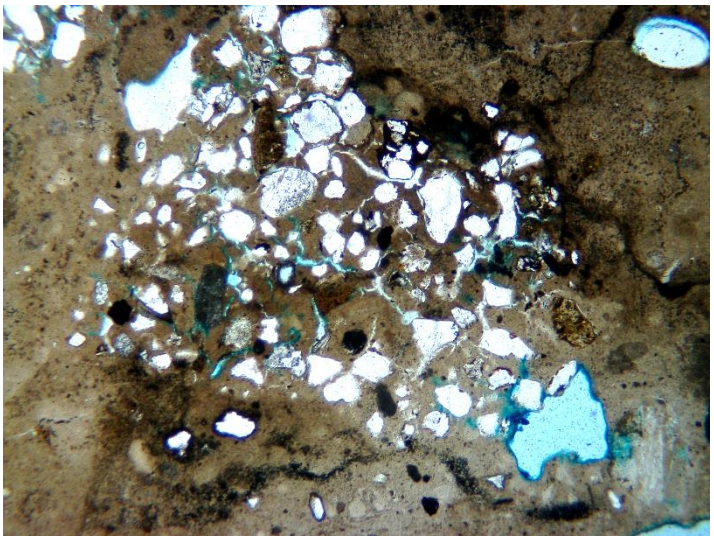


Plate No. 9:

A view in plane polarised light (ppl) of an aggregate rich partially calcined fragment of limestone. Its outer margin is highlighted by a dark rim, which is lost on the left side where it merges into the surrounding paste. The aggregates are of quartz and feldspar and do not show any evidence of reacting with the lime, though if temperature had been higher at time of calcining, there would have been the potential for reactions to produce hydraulic components.
Porosity and voids are highlighted by the blue dyed resin.
Field of view 2.4mm.

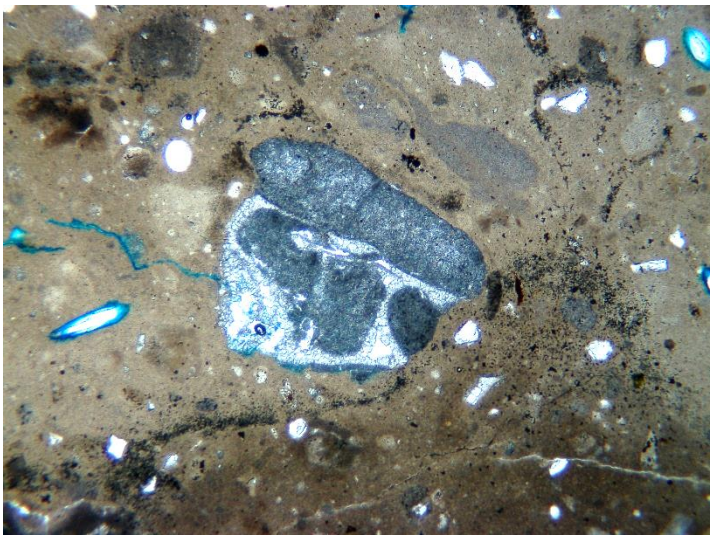


Plate No. 10:

Another view in plane polarised light, ppl, showing a limestone fragment, in the centre of the plate, which shows no evidence of alteration or burning, and this had perhaps been from the core of a lightly calcined fragment or incorporated from an area of unburnt limestone.
The paste in the upper part of the slide is partially carbonated whereas that in the lower part is uncarbonated.
Porosity and voids are highlighted by the blue dyed resin.
Field of view 1.2mm.

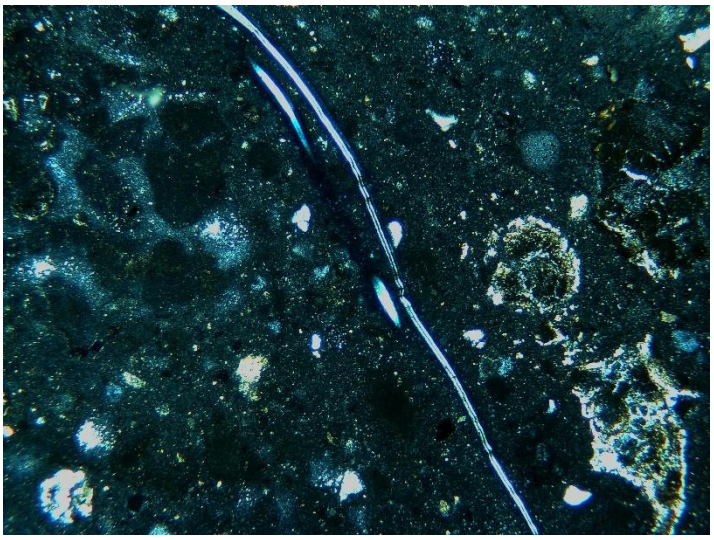


Plate No. 11:

A view in cross polarised light, xpl, of an area of the paste that shows minimal carbonation. The white speckles seen distributed throughout the slide are carbonated grains of lime, and are either fine limestone dust or carbonated Portlandite. Uncarbonated paste appears opaque in xpl. Two hair fibres transect the slide from upper left to lower right. Fragments of unburnt limestone can be seen in the lower right.
Porosity, voids, impregnating resin and opaque minerals all show dark in xpl.
Field of view 1.2mm

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 plaster prepared by Nigel Copsey of the Earth, Stone and Lime Company, which was received in CMC's Stirling Laboratory on the 8th April 2021, and identified as plaster from a quicklime trial using a local Oolitic limestone.

W A Revie
For CMC Ltd.

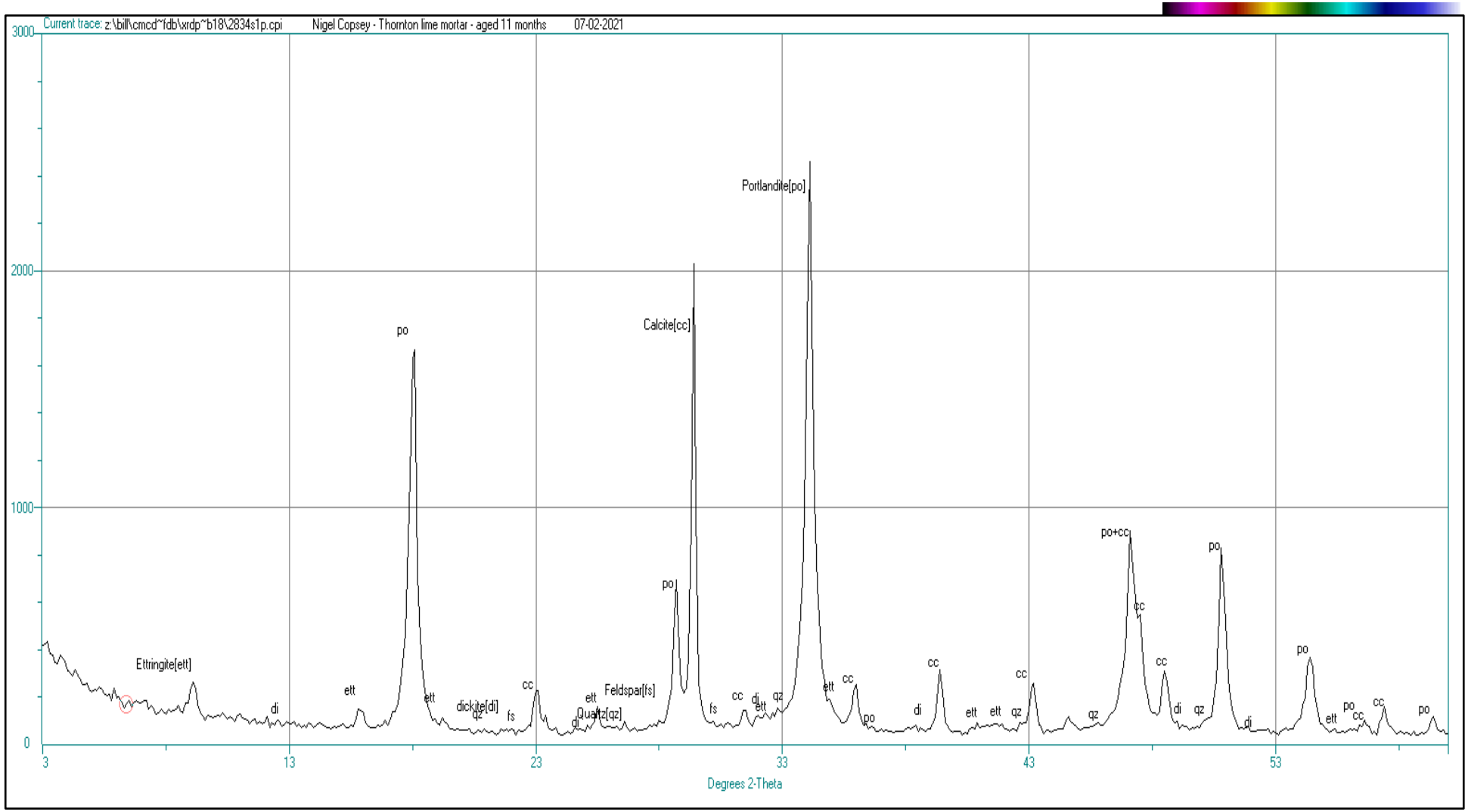


Figure No. 1: Sample SR2834-S1 – Binder from plaster made at Hall Farm, Maltongate.