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Our Ref: M/2088/20/C2-rev 1
Your Ref.: Oyster Shell Lime

14th February 2021

CERTIFICATE OF TEST & ANALYSIS ON OYSTER SHELL & OYSTER SHELL LIME

Project Reference	:	Oyster Shell Lime Mortar Research
Sample Source	:	Submitted by Atle Ove Martinussen
Sample Description	:	Oyster Shell and Oyster Shell Quicklime.
Date Received	:	2nd November 2020
CMC Sample Ref	:	SR 2813a (Oyster Shell) and SR 2813b (Quicklime)
Method of Test	:	Mineral composition by XRD analysis, with elemental analysis by XRF and thin section examination of shell

Samples

Following an emailed enquiry from Atle Ove Martinussen, on the 7th October 2020, relating to the testing of Oyster shell lime mortars, and subsequent email correspondence, a batch of samples were received in CMC's Stirling laboratory on the 2nd November 2020. Those received were identified as samples of the Oyster shell used in the production of quicklime and a sample of the Oyster Shell Quicklime, of the form used in the research being undertaken into the production of Oyster Shell Lime Mortars.

The samples were submitted for examination and analysed to provide information that may be of value in assessing the results from the testing of Oyster Shell Lime Mortars. With the specific requirement to determine if the lime produced from the shell would display any hydraulicity that that could influence the performance of the mortar produced.

Instruction to carry out an analysis and testing programme was received following a number of email exchanges over the period 7th October to 11th November 2020 with Alt-Ove Martinussen, Project Leader, for the research programme into the burning of Pacific Oyster Shell for the production of lime.

On receipt in the laboratory, the sample details were entered into the sample register and the unique sample identification number SR2813 allocated. The details of the samples received are presented below:

CMC Ref.	Client Ref	Sample Detail Supplied (Information on Sample Bags)
SR2813 – a	Sample 1	Pacific Oyster Shell from the West Coast of Norway, Typical of those used in burning trials
SR2813 – b	Sample 2	Oyster Shell Quicklime, from burning trials.

The samples, as received are shown in the following plates:


Plates No. 1 & 2: The above plates show the Oyster shell samples, as received, with the left plate showing the outer surface and the right plate the inner surfaces. Most of the shells retained varying amounts of organic and particulate matter adhering to their inner and outer surfaces.


Plates No. 3 & 4: The left plate shows the inner surface of a shell with starfish or sea stars (star-shaped Echinoderms) adhering to the surface, which was common to several shells in the sample received. Whereas, the right plate shows the outer surface with soiling and barnacles adhering to the surface, again not an uncommon occurrence. Note the layering of the shell, within which fine particulate matter can be seen.


Plates No. 5 & 6: The above images show the condition of the quicklime received, with the left plate showing the outer surfaces, and the right plate the inner surfaces. Note that the soiling and other features observed on the shell before calcining are mostly still apparent after calcining.

Plates No. 7 & 8: Close-up images of the Oyster Shell after burning. Some separation of the layering in the shell was apparent on some oyster shell pieces, with locally, particulate matter observed within these.

Method of Test

On receipt the samples were photographed, with their mass and size recorded. The samples were then submitted to an examination with the aid of a stereo-binocular microscope at a magnification up to x10.

To aid in the assessment of the form of binder that would be produced from the calcining of the Oyster Shell, representative sub-samples were prepared from both a sample of the raw shell ,and the quicklime, for analysis by X-ray Diffraction (XRD). This to permit identification of the mineral/crystalline components present.

As a further check, a representative composite sample of the Oyster shell was prepared with this again analysed by XRD. The sample used in the XRD analysis was then ignited in a muffle furnace at 975˚C for two hours, with the loss on ignition determined. The resultant quicklime was also analysed by XRD to ascertain if there were any hydraulic components present.

In addition, a sub-sample, of the powdered shell that was prepared for the XRD, was submitted to elemental analysis, in the form of their major oxides, by X-ray Fluorescence Spectrometry (XRF). The results of which would permit calculation of the Hydraulicity and Cementitious indices, to give an indication of any potential hydraulicity in the lime produced.

Results of XRD Analysis

Representative samples of the Oyster Shell and the Oyster Shell Quicklime were prepared by crushing them and grinding the materials in an agate mortar and pestle until all of the materials passed a 63µm sieve. A further composite sample, from several shell fragments was similarly prepared and analysed by XRD, both before and after igniting at 975˚C.

The individually prepared powdered samples were then backpacked into proprietary sample holders in preparation for presentation in the diffractometer. With the samples 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°. With the results of the XRD analysis presented in the following Figures, in the form of labelled X-ray Diffractograms:

Figure No. 1: Sample SR2813-L1 – Representative sample of Oyster Shell, as received.

Figure No. 2: Sample SR2813-L2 – Oyster Shell Quicklime, as received.

Figure No. 3: Sample SR2813-S1a – Oyster Shell, used in the loss on ignition determination and for XRF.

Figure No. 4: Sample SR2813-S1b – Calcined Oyster Shell from sample S1a above.

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

- **li** = Lime (Ca) Calcium Oxide, quicklime from the calcining of the Oyster Shell,
- **cc** = Calcite ($CaCO_3$) calcium carbonate, dominant form from which Oyster Shell is formed,
- **ar** = Aragonite ($CaCO_3$) another crystalline form of calcium carbonate, commonly found in shell,
- **be** = Belite (Ca_2SiO_4) *di*-Calcium Silicate, potential Hydraulic component, commonly found in hydraulic limes
- **qz** = Quartz (SiO_2) dominant component in most natural sands, and in some clays and silts,
- **an** = Anorthite, feldspar of the Plagioclase group, common rock forming minerals found in silts and clays,
- **ha** = Halite (NaCl), Sodium Chloride, from the Fjord water in which the Oyster had grown.

On the basis of the XRD analysis, it is confirmed that the Oyster shell contained trace amounts of quartz and feldspar minerals, which would infer that the oyster shell had most likely grown in an area where clays/silts were present in the waters. This is typical of what would be expected where rivers flow into the waters in which the Oyster nurseries were situated. On ignition, both in the kilns and in the laboratory furnace, the impurities within the shell structure had reacted with the lime producing trace proportions of hydraulic components in the form of Belite (Calcium silicate).

To permit quantification of the minerals/crystalline material present, the data from the XRD analysis was processed by Rietveld Refinement, in the Maud computer program. See results below:

Sample Ref.	**SR283-L1**	**SR2813-L2**	**SR2813-S1a**	**SR2813-S1b**
Form	**Oyster Shell**	**Quicklime**	**Oyster Shell**	**Quicklime**
	|--------As – Received----------|		**|----------Lab Produced----------|**	
Component	**Proportion (% by Mass)**			
Lime	-	98.0	-	95.8
Calcite	98.2	0.4	98.1	2.6
Aragonite	-	0.9	-	-
Belite	-	0.3	-	1.5
Quartz	0.3	-	0.9	-
Anorthite (Feldspar)	1.1	0.1	0.7	-
Halite (NaCl)	0.4	0.3	0.3	0.1
Total	**100.0**	**100.0**	**100.0**	**100.0**

From the above it was confirmed that the shell contained impurities and that in response to their presence, and dependent on the temperature reached during calcining, there is the potential for the lime to display a weak (feeble) hydraulic property.

The Chloride present is from the Fjord water in which the shell had grown.

To provide a measure of the potential hydraulicity of the lime, a sample of the shell was submitted to analysis by XRF to permit quantification of the major oxides present, which would permit the possible degree of hydraulicity to be calculated.

Analysis by X-Ray Fluorescence Spectrometry

The powdered sample prepared for XRD analysis was quartered and a representative sub-sample was pressed into a pellet in preparation for analysis by XRF. This analysis was carried out on our behalf by X-Ray Mineral Services Ltd. of Colwyn Bay, North Wales.

The results obtained from the analysis are presented below, along with the results of a Loss on Ignition determination, which was to allow the results to be normalised.

Sample Ref.	**SR2813-S1a**
Major Oxide	**% by Mass of Sample**
CaO	54.05
SiO_2	0.57
Al_2O_3	0.07
Fe_2O_3	0.04
MgO	0.25
MnO	<0.01
TiO_2	<0.01
K_2O	0.03
Na_2O	0.19
SO_3	0.36
P_2O_5	0.10
SrO	0.11
BaO	<0.01
LoI	44.20
Total	**100.00**

From the results obtained, the presence of Silica (Quartz) and the Aluminium (from the Feldspar,) it is indicated that the lime binder produced from the calcining of the Oyster Shell may display a very feeble degree of hydraulicity, albeit this may not be discernible in the mortar produced. However, to confirm the potential degree of Hydraulicity in the binder, the results from the XRF analysis were evaluated using following formulae:

$$\textbf{Cementation Index} = \frac{(SiO_2 \times 2.8) + (Al_2O_3 \times 1.1) + (Fe_2O_3 \times 0.7)}{(CaO + (MgO \times 1.4)}$$

$$\textbf{Hydraulicity Index} = \frac{(SiO_2 + Al_2O_3 + Fe_2O_3)}{CaO}$$

Sample Ref.	**Hydraulicity Index**	**Cementation Index**
SR2813-S1a	0.01	1.48

The following is an example of the guidance given historically, for the interpretation of the values above:

Indices	**Hydraulicity**	**Cementation**
High calcium lime (air lime)	<0.10	<0.30
Feebly Hydraulic	0.10 to 0.20	0.30 to 0.5
Moderately Hydraulic		0.50 to 0.70
Eminently Hydraulic	0.20 to 0.40	0.70 to 1.10

Based on the above it is indicated that the lime produced from the calcining of the Pacific Oyster Shell, from the West coast of Norway, would effectively produce a **non-hydraulic**, high calcium air lime.

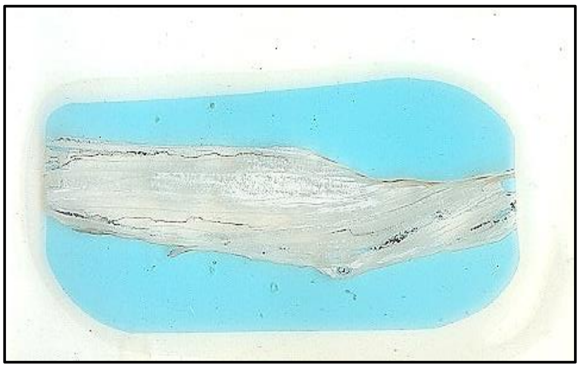
Notes:

The Hydraulicity Index, calculated from the sum of the SiO_2+Al_2O_3+Fe_2O_3 divided by the total CaO, has inherent defects, as there is no allowance for the magnesia commonly present in the raw materials used in the production of limes and cements from Limestone, and it is based on the supposition that the silica and alumina are quantitatively interchangeable, which results in it not being an ideal method in the assessment of certain classes of hydraulic limes and cement. Hence, the Cementation Index was substituted, and used historically for this classification purposes.

The Cementation index can, however, be used in quantifying and assessing the hydraulicity of a cementitious binder, whether it is a Hydraulic lime, Natural cement or Portland cement, but only if the effect of the calcining temperature, and other processing variations, are also taken into consideration. Therefore, when used purely comparatively on products of the same genre, the indices can be a useful aid in assessing the potential performance and variability of a particular product, but it is not suitable for assessing compliance of a particular hydraulic product, with a specification. However, in this particular application it could only be used comparatively, and, then, with caution.

Microscopic Examination

A thin section was prepared from one of the shell pieces for examination in the polarised light microscope.

Plates No. 9 & 10: The above plates show the section of shell from which the thin section was prepared, with this shown in the left plate, with the prepared thin section shown in the right plate.

The purpose of the examination was to determine if the quartz and feldspar was present purely as adhering surface material, or whether it was encapsulated within the fabric of the shell, trapped within the lattice as the shell formed.

The following plates show examples of the fabric, in which fine silt sized particles were observed both within the lenses formed as the shell grew, and bonded to the outer surface.

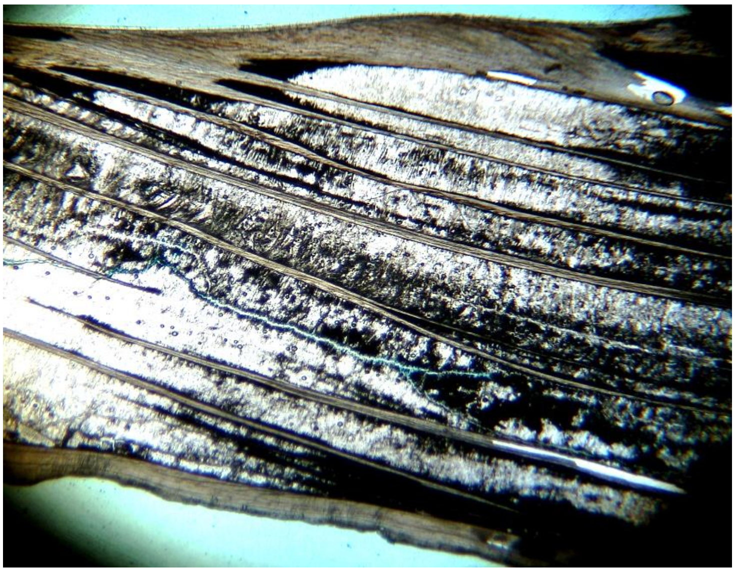


Plate No. 11:

View through the thickness of the shell, in plane polarised light (ppl).

The view shows the structure of the shell with calcite forming the majority of the materials present, along with minor silt sized grains observed within the fabric.

Porosity is highlighted by the blue dyed encapsulating resin
Field of view 10mm

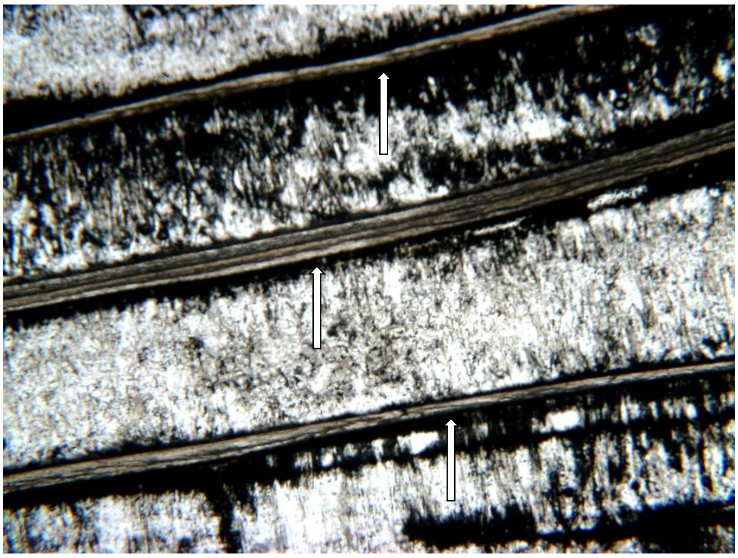


Plate No. 12:

A magnified view through the thickness of the shell, in ppl.
Here the thin dense slow growth laminae (arrowed) can be seen interspersed between the faster grown, wider laminae, formed during warm weather, growth periods.

Dark amorphous material can be seen interspersed throughout both the upper and lower rapid growth zones.

Porosity is highlighted by the blue dyed encapsulating resin
Field of view 3.5mm

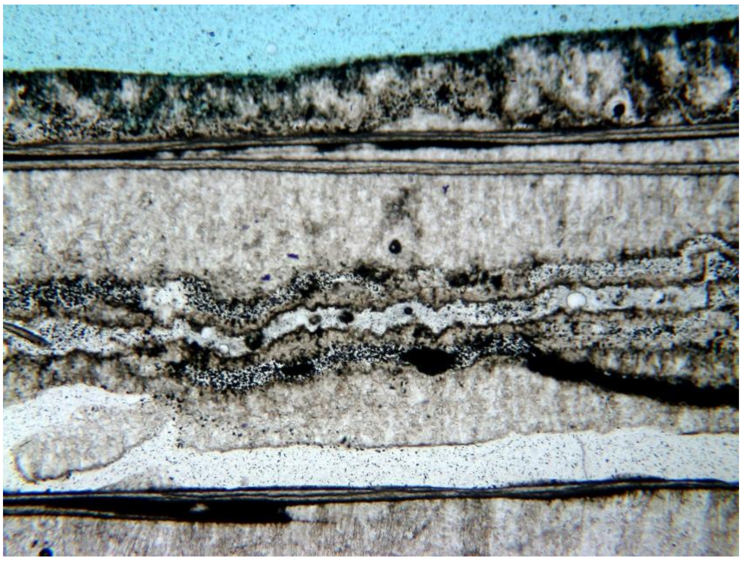


Plate No. 13:

View through the thickness of the shell, in ppl.
This section contains a crack through the centre of a fast growth zone, centre of plate, through which water appears to have percolated with the deposition of fine particulate matter, encapsulated in redeposited calcite. Both quartz and feldspar are noted, along with fine indeterminate amorphous material.

Porosity is highlighted by the blue dyed encapsulating resin
Field of view 0.8mm

Summary

On the basis of the examination and analysis carried out on the samples of quicklime and oyster shell received it can be confirmed that the shell is suitable for the production of a High Calcium air lime, with the minor contaminants present insufficient to result in the production of hydraulic components in sufficient quantity to impart any degree of hydraulicity to the lime, when the shell is calcined at a temperature high enough for them to form.

Although it cannot be classed as a representative sample, the sample calcined in the laboratory was analysed by the methods detailed in EN 459 Part 2: 2010, and on the basis of the results it is indicated that the shell received, if representative of that used in the site calcining trials, can produce a quicklime of sufficient quality to comply with the requirements of EN 459 Part 1: 2015, with respect to a grade CL90, with regard to its chemical composition:

However, there was insufficient material to carry out the physical tests, albeit the results would not have been applicable, as ideally, these should be carried out on freshly produced material sampled from the kiln product.

Notwithstanding, as it is understood that the lime is being produced from an indigenous material, i.e., Pacific Oyster from the West coast of Norway, which are to be calcined following traditional techniques, i.e., in a vertical batch kiln, fired using wood as the fuel, it was clearly demonstrated that a suitable quicklime can be produced.

As the product is to be used in the repair and conservation of traditional buildings, in the form of a lime binder for use in mortar, plaster, render and limewash, it is considered that the oyster shell lime produced, as demonstrated by the samples submitted, provided adequate control of the calcining process is exercised, will provide an ideal binder for its intended purpose.

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 received in CMC Ltd.'s laboratory on the 2nd November 2020, from Atle Ove Martinussen, which were identified as Oyster Shell and Oyster Shell Quicklime.

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For CMC Ltd.

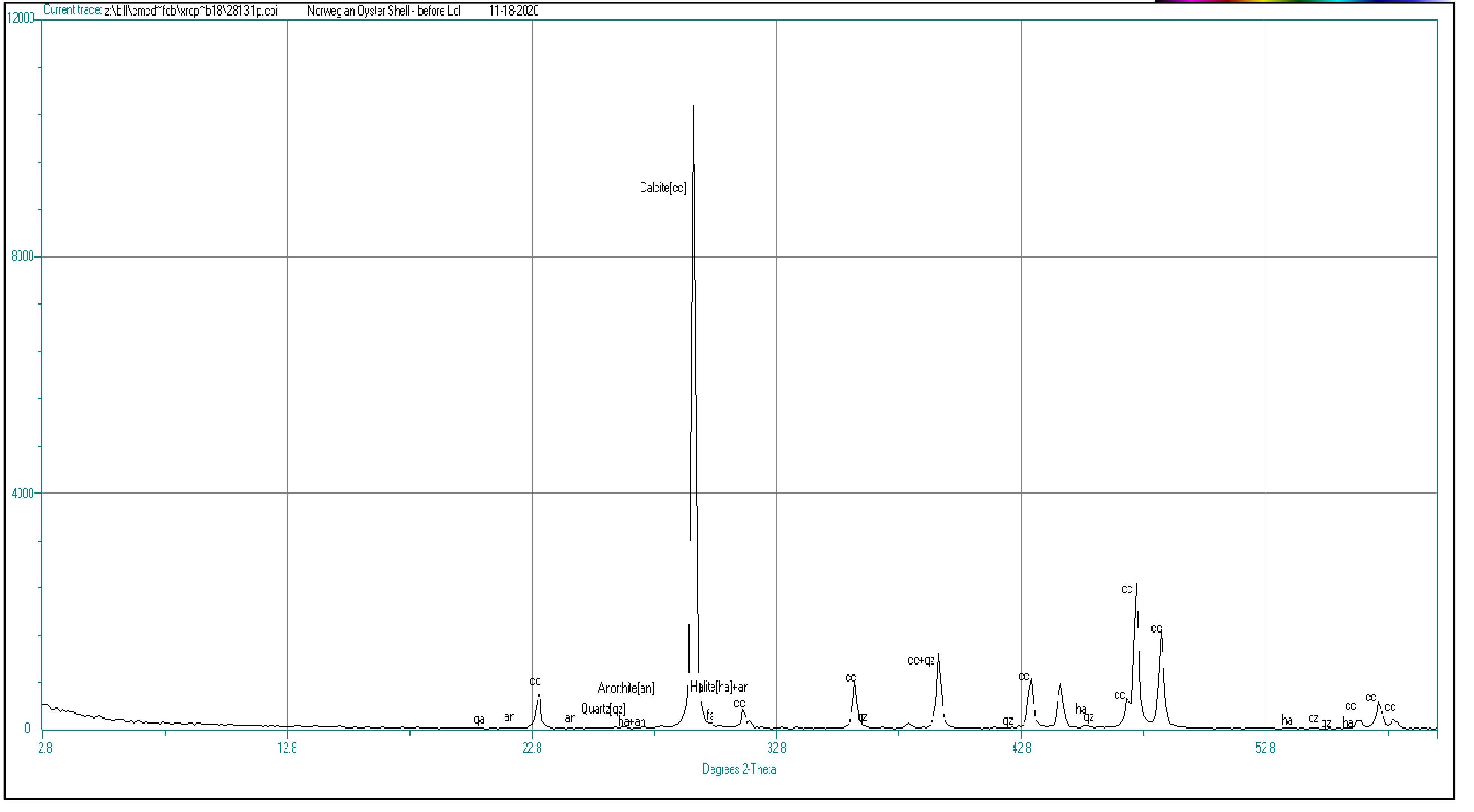

Figure No. 1: Sample SR2813-L1 – Representative sample of Oyster Shell, as received.

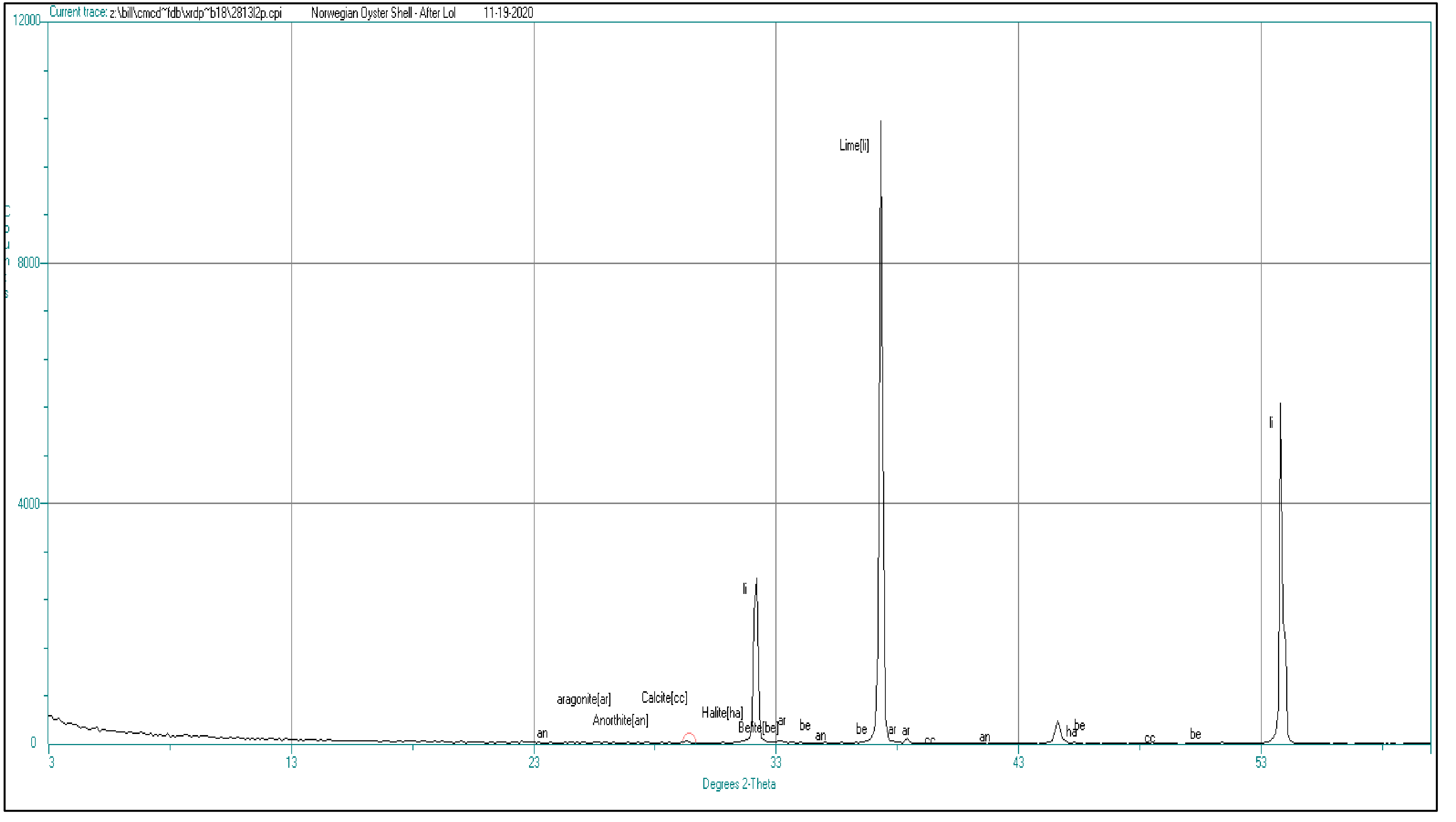

Figure No. 2: Sample SR2813-L2 – Oyster Shell Quicklime, as received.

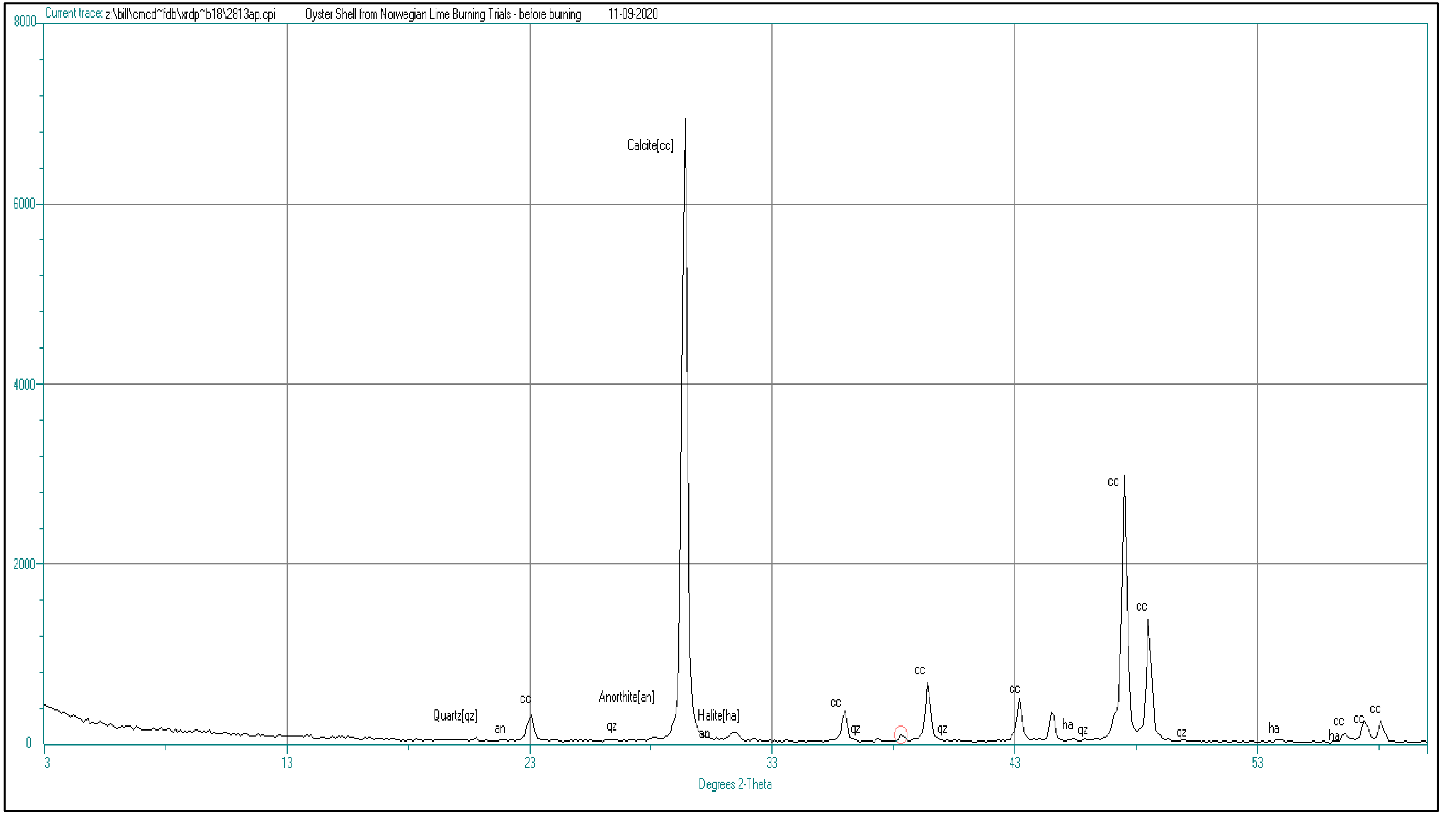

Figure No. 3: Sample SR2813-S1a – Oyster Shell, as used in the loss on ignition determination and for XRF.

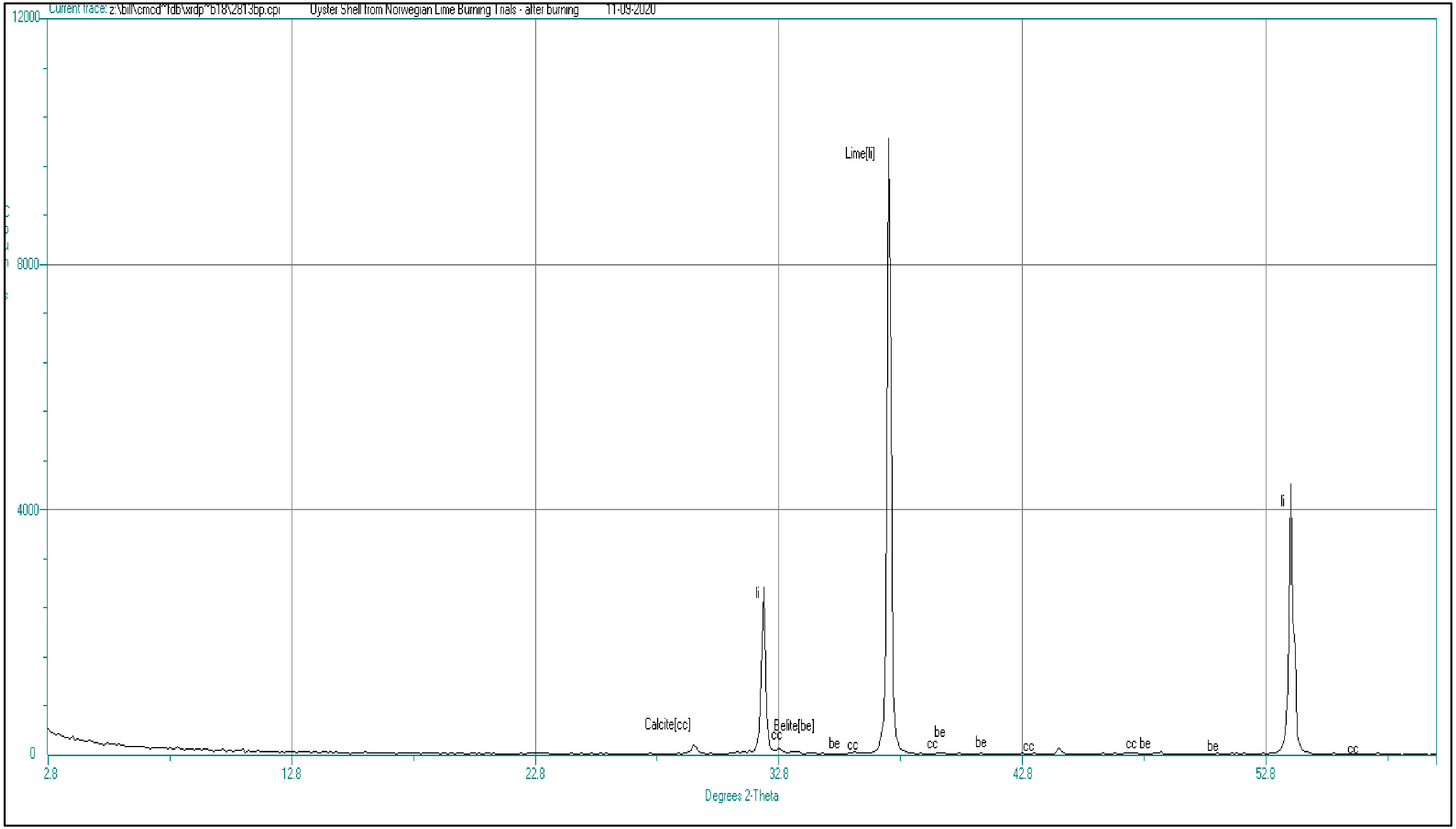

Figure No. 4: Sample SR2813-S1b – Calcined Oyster Shell from sample S1a above.