# Manuscript Details

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| **Manuscript number** | JOLT_2017_242_R1 |
| **Title** | Micro-Raman spectroscopy for the characterization of rock-art pigments from Abrigo del Águila (Badajoz – Spain) |
| **Article type** | Research Paper |

**Abstract**

Micro-Raman spectroscopic technique allowed the characterization of organic and inorganic pigments of different colours sampled from a rock-art shelter named Abrigo del Aguila, located in the district of Badajoz, Cabeza del Buey (Extremadura – Spain). Micro-Raman analyses has been coupled with SEM observation and elemental analyses (EDS). The white and the black colours, used for non-representative figures, have been identified respectively as anatase and amorphous carbon, while two different type of red pigment has been found on figurative representations. The darker one, sampled, from a sun-figure, comprises an indeterminate organic compound beside of hematite. The second one, sampled from an anthropomorphic figure, is of a brilliant red and only hematite has been recognized in it.

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| **Keywords** | Laser spectroscopy; Micro-Raman; Scanning Electron Microscopy; Schematic rock-art; Pigments. |
| **Manuscript category** | Spectroscopy |
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## Submission Files Included in this PDF

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Response to reviewer.docx [Response to Reviewers]

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Cover Letter

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The authors confirming that it is of original material i.e. has not been submitted elsewhere for publication.

This research has been carried out in, with an interdisciplinary team from Italy (TekneHub Laboratory - Ferrara University), Portugal (Geosciences Centre) and Spain (ACINEP).

This paper explores the processes involved in the production of prehistoric paintings using red, white and black pigments; from sourcing the raw material to the processing of pigments.

In this work we present the results of micro-Raman spectroscopy analyses on pigments samples of a site in Badajoz Spain. Pigments from these rock painting panels' as well natural ochre samples were analysed using Raman spectroscopy, Scanning Electron microscopy (SEM). Results from red pigments revealed that the main chemical element was iron (Fe). The Raman spectra indicated the presence of hematite.

With SEM analyses, other chemical elements besides iron were identified; the white and the black colours, used for non-representative figures, have been identified respectively as anatase and amorphous carbon, while two different type of red pigment has been found on figurative representations. The darker one, sampled, from a solar figure, comprises an indeterminate organic compound beside of hematite. The second one, sampled from an anthropomorphic figure, is of a brilliant red and only hematite has been recognized in it. Possibility of rock-art dating using carbon trace from black figures is also discussed.

Dear Dr. Dahotre,

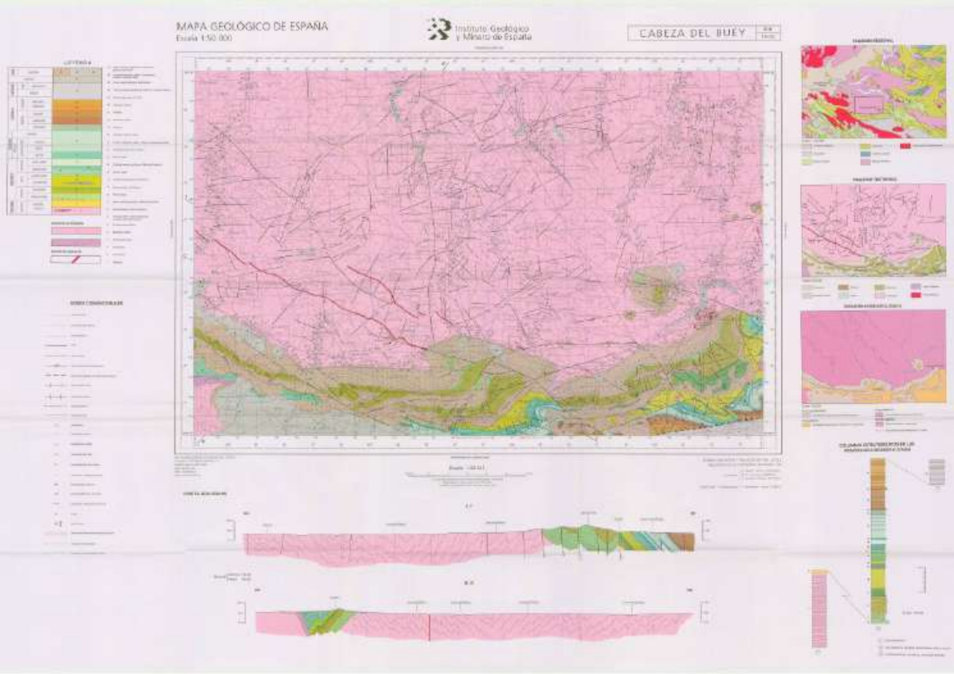
Thank you for considering our article for publication in Optics and Laser Technology. We are grateful also to the reviewer for the valuable suggestions provided, which were of great help in revising the manuscript.

Here are responses to the reviewer comments:

Comment 1: What type of rocks is supporting the paintings? Are they calcareous, sandstone, granitic… etc.? It is important to know their composition in order to distinguish between pictorial and supporting materials. Samples from these rocks should have been analyzed.

***(Lines 29-30) The archaeological site of Abrigo del Águila is placed in the East part of the quarzitic alignment of Sierra de la Rinconada (Fig. 1).***

*Even if it is no more possible to access the site and collect samples of the supporting rocks to be analysed, it's possible to state that the substrate of the paintings is quartzite, on the base of the geological map of the area (also available on line at . http://info.igme.es/cartografiadigital/geologica/Magna50Hoja.aspx?intranet=false&id=806)*



Comment 2: The Raman spectrum shown in Fig. 4 does not correspond to reference spectra of natural alizarin (e.g. Ref. 18, Table 2, Az powder, the strong bands at 1569, 1191 and 470 cm-1 are not present in the spectrum of Fig. 4; see also the spectra from Pozzi et al., Heritage Science, 1 (2013) 23). The additional record of the spectral range 2800-3200 cm-1 would have shown relevant C-H aromatic stretching bands of the anthraquinone rings at wavenumbers higher than 3000 cm-1 (or the presence of C-H stretching bands below 3000 cm-1 of possible saturated organic compounds). Moreover, several organic compounds are present in madder root. Ruberthryrine from the root dried, fermented and treated with acids yields xylose, glucose, alizarin and purpurin. Did the prehistoric painters know this technique? This is the first time that alizarin is considered a prehistoric pigment. Clear evidences must be presented to confirm this finding, i.e. a reliable assignment of the Raman bands observed in the spectrum attributed to alizarin. Is the spectrum of Fig. 4 indicating a mixture of different compounds from madder root? Alizarin is a very fluorescent compound exciting at 632 nm. A higher wavelength, 785 nm (Ref. 18) or 1064 nm is required to avoid the

strong background of fluorescence radiation, and the use of the SERS technique to get acceptable Raman spectra.

***(Lines 119-147) Ochre are not the only raw material used to make red pigment. The analysis of the sample taken from the sun-figure at Abrigo del Águila rock-art shelter suggests the use of an organic component in association with hematite. This figure is of a dark shade of red if compared with other red figures in the painted shelter and Raman analysis of the sample taken from it has revealed the presence of undefined organic matter with peaks at 1176, 1232, 1359 (vs), 1371, 1484, 1590.(Fig.4).***

***This organic matter may or may be not part of original pigment.***

***Organic chromophores, although referenced since historical times, are very difficult to preserve, so they are sometimes identified as dyes but not used as pigments. The extraction of the dyes has been done from different parts of the plants: in some the leaves were used, while others used flowers, roots, fruits, trunks or seeds. The dyes could be extracted through complex processes involving different operations, such as maceration, distillation, fermentation, decantation, precipitation, filtration, etc. [17].***

***A widely used vegetable dye was the natural indigo known from the Egyptians to the Britons, extracted from the Isatis tinctoria plant [18]. From the garança plant (Rubia tinctorium) various colouring compounds were extracted, such as alizarin and purpurin (not to be confused with purpurin of animal origin). These substances have been used at least since the 3rd millennium BC to dye tissues and still been used and marketed today [19,20]. Alizarin is a dye molecule of the anthraquinone family, which can be found in many other plants and animals [21, 22]. . Some examples of anthraquinone used as pigments in an archaeological context are cited in Clementi et al. (2011) [21].***

***Because of weak Raman signal and strong fluorescence background, alizarin and other highly fluorescent antraquinone dyes are usually detected using surface-enhanced Raman scattering (SERS) in order to improve Raman signal and quenched the fluorescence background [23, 24].***

***(Line147) Moreover, there is the possibility that the organic compound derives from pigment's rearrangement.***

Comment 3: On the other side alizarin is photodegradable and partially soluble in water. Does alizarin support thousands of years on the wall of a rock shelter with sunlight and possible humidity and rain?

***There´s some bibliographic reference on organic compounds recognized in rock art (Serrano et al., 2007; Clementi et al., 2011).***

Comment 4: Check if the spectrum shown in Fig. 4 does not correspond to a prehistoric pigment, but to organic compounds from biofilms covering the pictograph (microbial photo-pigments, bacteria, fungi, algae or lichen colonies… etc.).

***(Lines 143-146) On the other hand, many of the organic materials identified are "concretions" that may appear for biogenic or climatological reasons, associated to the presence of microorganisms that, in contact with the humidity and depending of the temperature conditions and sun exposure, mineralize - biomineralization [25], making it difficult to interpret the obtained results.***

We would be happy to make any further changes that may be required.

## Highlights

In this paper we present the chemical results from rock painting of Abrigo del Águila rock shelter.

Nine samples of pigments of white, red and black pigments samples were collected.

The analyses of the samples have been carried out using Raman microspectroscopy and SEM-EDS chemical analyses.

Results indicate presence of titanium (anatase), hematite, charcoal and a possible organic substance.

# Micro-Raman spectroscopy for the characterization of rock-art pigments from *Abrigo del Águila* (Badajoz – Spain)

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## ABSTRACT

Micro-Raman spectroscopic technique allowed the characterization of organic and inorganic pigments of different colours sampled from a rock-art shelter named Abrigo del Aguila, located in the district of Badajoz, Cabeza del Buey (Extremadura – Spain). Micro-Raman analyses has been coupled with SEM observation and elemental analyses (EDS). The white and the black colours, used for non-representative figures, have been identified respectively as anatase and amorphous carbon, while two different type of red pigment has been found on figurative representations. The darker one, sampled, from a sun-figure, comprises an indeterminate organic compound beside of hematite. The second one, sampled from an anthropomorphic figure, is of a brilliant red and only hematite has been recognized in it.

KEYWORDS: Laser spectroscopy, Micro-Raman, Scanning Electron Microscopy, Schematic rock-art, pigments.

## 1. INTRODUCTION

As confirmed by several previous studies, μ-Raman spectroscopy is proving to be a valuable tool for the investigation of pigments used in prehistoric rock-art paintings as well as on a wide range of archaeological artefacts [1-5]. In this work Raman spectroscopy and SEM-EDS analysis have been carried out in order to characterize rock-art pigments from *Abrigo del Aguila* (Cabeza del Buey, Badajoz, Spain).

### 1.1 Regional and Geological Settings

The archaeological site of Abrigo del Águila is placed in the East part of the quarzitic alignment of *Sierra de la Rinconada* (Fig. 1). The site is located 40 meters above soil level, within a great rock crack of about meters of length. Since prehistory this place have played an important role for its orographic characteristics as it´s located in the region which separates *Sierra de la Rinconada* from *Sierra de la Osa*, becoming an inevitable passage for the routes towards the prairies of *Sierra de la Rinconada*.

Fig. 1 - Geographic Location of *Abrigo del Águila* (Badajoz, Spain)

### 1.2 Rock art pictographs at *Abrigo del Águila*

The present paper classifies and investigates rock-art pigments from the archaeological site of *Abrigo del Águila*. The pictorial rock-art group consists of thousands of figures that are arranged in

45 rock panels and are placed in the inner part of the rock-shelter. Preliminary studies revealed repeated pictorial motives that are irregularly spread on the surface of the archaeological site: some of the areas are characterized by few motives, while other show real palimpsests. Motifs are both abstract and figurative.

Despite the high concentration of these figures, it is difficult to evaluate the figurative overlapping in order to determine the diachronic evolution of the graphical use of this site. Even if it is not possible to establish a detailed chronology, cultural references exist that allow to refer the site to the lap-time between the end of the Neolithic and the Copper Age [6].

In schematic rock-art of Iberian Peninsula figures are usually of a red colour, drown with mineral pigments rich in iron oxides, such as hematite or goethite. Black and white pigments are, instead, uncommon [7]. At *Abrigo del Águila* rock-art shelter most of the figures are red, but white and black representation are also present. The red colour is of several shades, from orange tones to darker ones. The different colour shade could be due to several factors such as conservative state, environmental and biological degradation, interaction with rock surface, diverse adherence to the rock support, different thickness of pigments (Fig.2).

Fig.2 - Overlapping figures in panel 36, from *Abrigo del Águila*.

## 2. MATERIALS AND METHODS

### *2.1 Sample collection*

Nine samples were collected in strategic figurative representations, in order to encompass all the chrono-cultural spectrum of the painting motives and variation of the colour of the figures (white, black and various shades of red) were also taken into account. Where possible, sample collection has been done using non-contact ethical extraction techniques (applying the code of ethics and guidelines for practice of American Institute for conservation). Each sample, weighing between 10 and 100 mg, was extracted in areas of the panel where pigment was easily observed. Each sample was obtained using a sterilized tungsten scalpel and insert in a 0,5ml microcentrifuge tube [8].

The archaeometric research aims to create knowledge based with all the relevant parameters and can provide reliable data for interpretation, conservation and management of rock-art sites. In this context, analytical techniques applied to elemental, chemical and structural characterization of parietal pigments and deterioration causes, thus provide vital information.

### *2.2 Instrumental*

Micro-Raman spectroscopy was employed to determine the mineralogical composition of pigment samples. Raman measurements were performed by LabRam HR800 spectrometer (Horiba Jobin Yvon, France), coupled with an Olympus BXFM optical microscope (Olympus, Tokyo, Japan). The spectrometer was equipped with air-cooled CCD detector (1024 × 256 pixels) set at –70 C°, and with 600 and 1800 grooves/mm gratings. The laser beam was concentrated in a spot with 1 mm diameter and the spectral resolution was approximately 4 $cm^{-1}$. The He-Ne laser line at 632.82 nm was used as excitation source and was filtered to keep the laser power varying from 0.2 to 10mW.

Exposure time, beam power and accumulations were optimized for each sample in order to obtain sufficiently informative spectra but ensuring to avoid alteration of the sample. Several measurements were performed at low powers and increasing it gradually, where possible. Raman spectra were recorded in the range of 200–2000 $cm^{-1}$ with an exposure time of 5–16 seconds and 5-11 accumulations. The 10x and 50 x microscope objectives were employed to focus the laser beam onto the samples, placed on the X-Y motorized sample holder, and the spot size diameter was about 2–3µm. The wavelength scale was calibrated using a Silicon standard (520.5 $cm^{-1}$) and the acquired spectra were compared with scientific published data and reference databases, such as Horiba LabSpec 5 (Horiba) and RRUFF (RRUFF, University of Arizona, AZ, USA).

SEM-EDS analysis has been carried out in order to study the morphology of pigment particles and to determine the chemical composition of each sample. SEM observations and EDS microanalyses were carried out using a ZEISS EVO MA15-HR scanning electron microscope (SEM) (Zeiss, Jena, Germany), equipped by Oxford Inca 250 X-Act EDS microanalysis system (Oxford Instruments, Abingdon, United Kingdom). The specimens were not coated with gold or carbon; therefore, the analysis was performed under variable pressure. The combined use of secondary and backscattered electron imaging modes provided to better recognize the pictorial layers from the rock substrate. The analyses were performed using an accelerating voltage of 20 kV and a working distance of 8.5 mm.

## 3. RESULTS AND DISCUSSION

Table 1- Results

### *3.1 Red pigment*

Red figures at *Abrigo del Águila* rock-art shelter are mainly composed of hematite. Raman spectra of the red samples are presented in Fig. 3 and show the typical peaks of this mineral at 222, 289, 406, 607, 658, 1313 $cm^{-1}$. This is consistent with the literature about prehistoric rock-art pigments and with the results obtained by other authors [1, 3, 8-13]. Hematite is a very common mineral and is one of the principal components of natural ochres that have been used since prehistory in mural paintings [14].

What is remarkable in the spectra obtained in this research is the double peak at 607 and 658 $cm^{-1}$. These peaks can be attributable to a disordered phase of hematite, as occur in natural ochre [13, 15] but many other factors, both anthropic and natural, such as heating, grinding, biodegradation and weathering can be responsible for the arising of these peaks and Raman technique cannot be used to differentiate among them [15].

Figure 3 - Raman spectrum of hematite from sample AGU_05.

In addition to high concentration of Fe, SEM-EDS analyses on red samples revealed also the presence of Al and Si. Thanks to the thickness of the pictorial layer, it is possible to attribute these elements to the pictorial matrix and not to the rock substratum, reinforcing the hypothesis that red pigment has been obtained from natural red ochre [16].

Table 2 - Semi-quantitative SEM-EDS analyses performed on two different type of red pigment (AGU_05/AGU_09)

Ochre are not the only raw material used to make red pigment. The analysis of the sample taken from the sun-figure at Abrigo del Águila rock-art shelter suggests the use of an organic component in association with hematite. This figure is of a dark shade of red if compared with other red figures in the painted shelter and Raman analysis of the sample taken from it has revealed the presence of undefined organic matter with peaks at 1176, 1232, 1359 (vs), 1371, 1484, 1590.(Fig.4).

This organic matter may or may be not part of original pigment.

Organic chromophores, although referenced since historical times, are very difficult to preserve, so they are sometimes identified as dyes but not used as pigments. The extraction of the dyes has been done from different parts of the plants: in some the leaves were used, while others used flowers, roots, fruits, trunks or seeds. The dyes could be extracted through complex processes involving different operations, such as maceration, distillation, fermentation, decantation, precipitation, filtration, etc. [17].

A widely used vegetable dye was the natural indigo known from the Egyptians to the Britons, extracted from the Isatis tinctoria plant [18]. From the garança plant (Rubia tinctorium) various colouring compounds were extracted, such as alizarin and purpurin (not to be confused with purpurin of animal origin). These substances have been used at least since the 3rd millennium BC to dye tissues and still been used and marketed today [19,20]. Alizarin is a dye molecule of the anthraquinone family, which can be found in many other plants and animals [21, 22]. . Some examples of anthraquinone used as pigments in an archaeological context are cited in Clementi et al. (2011) [21].

Figure 4 - Raman spectrum of sample AGU_ 09 (organic substance)

Because of weak Raman signal and strong fluorescence background, alizarin and other highly fluorescent antraquinone dyes are usually detected using surface-enhanced Raman scattering (SERS) in order to improve Raman signal and quenched the fluorescence background [23, 24].

On the other hand, many of the organic materials identified are "concretions" that may appear for biogenic or climatological reasons, associated to the presence of microorganisms that, in contact with the humidity and depending of the temperature conditions and sun exposure, mineralize - biomineralization [25], making it difficult to interpret the obtained results.

Moreover, there is the possibility that the organic compound derives from pigment's rearrangement.

### *3.2 White pigment*

Evidences of the use of white pigment in Spanish prehistoric rock art are very rare and so are the available informations in literature [7, 10] for this reason is very important to determine chemical and mineralogical composition of prehistoric white pigments.]. μ-Raman analyses performed on white pigment sampled from Abrigo del Águila rock art shelter has shown the presence of anatase (fig.5) with characteristic peaks at 143, 196, 396, 516 and 637 cm1 [29, 31]. This mineral is a polymorph of titanium dioxide and occurs in nature in many geological contexts but in relatively small quantities and rarely as well formed crystals [32].

Figure 5 - Raman spectrum of sample AGU_01 (anatase)

Commercially, anatase has started to be widely used as white colourant since the early twentieth century, due to the difficulties to synthetize it before the modern commercial manufacture began. For this reason anatase is considered one of the key inorganic pigment indicators in authenticity studies and some researchers assume its use in antiquity as unlikely. Nevertheless it has been found on pottery both from Neolithic and roman period as a component of the white pigment used for decorations [38, 32] and on rock-art pigments, as reported by Edwards et al. [33]. Laser spectroscopy techniques, such as μ-Raman spectroscopy, are able to detect anatase in clay white pigments, even if it is present in very small quantities, as anatase has an extraordinarily intense Raman scattering [23].

SEM-EDS analysis performed on the white pigments sampled at *Abrigo del Águila* rock-art shelter shows a widespread abundance of Al, Si and K, suggesting a clay composition (kaolinite, illite) of the pigment (Fig. 6).

The presence of punctual concentrations of titanium is also noticeable and is referable to the presence of titanium dioxide (anatase) in the matrix of the pigment.

Raman bands attributable to clay minerals have not been recognized in the spectra due to the high fluorescence background of aluminosilicates [11] and because the intense Raman scattering of anatase, whose bands dominate the spectrum [23]. Thus, contribution of SEM-EDS analysis has been essential to attempt any interpretation.

Figure 6 - SEM-EDS analyses on a sample of white pigment. High percentages of Ti are concentrated in the small white crystals attributable to anatase. The other elements, which were detected in the grey matrix, suggest clay minerals in the samples.

As anatase can be incorporated naturally in kaolin, usually at the 2% level, and the latter has been used as white pigment in antiquity [31, 33], we assume that the white figures at *Abrigo del Águila* rock-art shelter could have been made using minerals from the illite-kaolinite group. In particular, kaolin is found relatively pure in numerous location and requires little processing for use as a pigment [13].

### *3.3 Black pigment*

Black figures are also rare in Iberian Peninsula schematic *rock-art,* although are more frequent than the white ones. The most often detected raw material used for drawing black figure is amorphous carbon [3, 8, 11] and this result has been confirmed also in this case. Small black particles have been found in the specimen taken from the dark figure and analysed with Raman spectroscopy giving rise to amorphous carbon spectrum (Fig. 7).

Figure 7 - Raman spectrum of sample AGU_8 (amorphous carbon)

Carbonaceous materials (of mineral, vegetable or animal origin) used as pigment has been identified in prehistoric sites worldwide and carbon-based black pigments has been largely employed even later in times [13, 34]. μ-Raman spectroscopy has played and continues to play an important role for their study and characterization. Through this analytical technique is possible to differentiate between different type of carbonaceous material allowing for example provenance study or archaeological interpretation 34, 35].

Figure 7 shows the strong and broad G and D1 bands of amorphous carbon at 1603 and 1348 $cm^{-1}$ respectively [3, 4, 8, 11, 34] that are typical of disordered $sp^2$ carbon. In particular, D1 band at 1348 $cm^{-1}$ is one of the D bands associated with different causes of disorder [34]. The absence of bands in the 960-970 $cm^{-1}$ spectral region rule out the use of ivory or calcined bones in the pigment preparation, as previously observed by other authors [3, 4, 8, 11]. These observations suggest that a charcoal of vegetal origin or soot is the main component of the pigment used to draw the black figure at *Abrigo del Águila* rock-art shelter.

## 4. CONCLUDING REMARKS

Archaeometric analyses, carried out thanks to laser spectrometric techniques, allowed to characterise rock art pigments used at *Abrigo del Águila* in the Spanish region of Extremadura. In this context, red, white and black pigments were collected and analysed.

This is the first investigation on white pigment used in Extremadura rock art, but the use of white pigments rich in titanium has already been documented in other rock-art site in the Iberian Peninsula.

The red hematite pigments results have origin from various types of regional ochres. One of the samples revealed the presence of organic substance associated with hematite.

Black figures were drawn using amorphous carbon, the presence of carbonaceous material in the pigment could allow the use of radiocarbon dating (AMS) techniques [36] to obtain an absolute chronology of the rock-art at *Abrigo del Águila,* favouring further comparisons and interpretations of the archaeological context.

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| **SAMPLE** | **COLOUR** | **DESCRIPTION** | **RAMAN RESULTS** | **SEM-EDS RESULTS** |
|---|---|---|---|---|
| AGU_01 | White | Non-representative figure, parallel lines | Anatase | Titanium<br>Barium |
| AGU_02 | Red | Pectiniform | Hematite | -- |
| AGU_03 | Red | Eye Idol | Hematite | Silicium |
| AGU_04 | White | Non-representative figure, parallel lines | Hematite<br>Calcite | -- |
| AGU_05 | Red | Anthropomorphic figure | Hematite | Iron<br>Titanium |
| AGU_06 | Red | Red figure | Not analysed | Not analysed |
| AGU_07 | Red | Pectiniform figure | Hematite | -- |
| AGU_08 | Black | Non-representative figures, parallel lines | Amorphous carbon | Barium |
| AGU_09 | Dark red | Soliform figure | Hematite + Organic compound | Iron<br>Arsenic<br>Barium |

Table 1 - Results

| **Element** | **Red (AGU_05) Hematite** | **Dark Red (AGU_09) Organic Substance** |
|---|---|---|
| | Weight% | Weight% |
| C | 11.64 | 4.83 |
| O | 43.13 | 40.62 |
| Na | 0.17 | 0.00 |
| Mg | 0.43 | 0.00 |
| Al | 4.84 | 3.21 |
| Si | 20.42 | 5.12 |
| P | 0.58 | 0.87 |
| S | 0.45 | 0.25 |
| K | 1.00 | 0.45 |
| Ca | 0.40 | 0.00 |
| Ti | 0.51 | 0.00 |
| Fe | 16.44 | 43.00 |
| As | 0.00 | 1.66 |

Table 2- Semi-quantitative SEM-EDS analyses performed on two different type of red pigment (AGU_05/AGU_09)

## List of Figures

Fig. 1- Geographic location of *Abrigo del Águila*.

Fig. 2- Overlapping figures in panel 36, from *Abrigo del Águila*.

Fig. 3- Raman spectrum of sample AGU_05 (hematite).

Fig. 4- Raman spectrum of sample AGU_ 09 (organic substance).

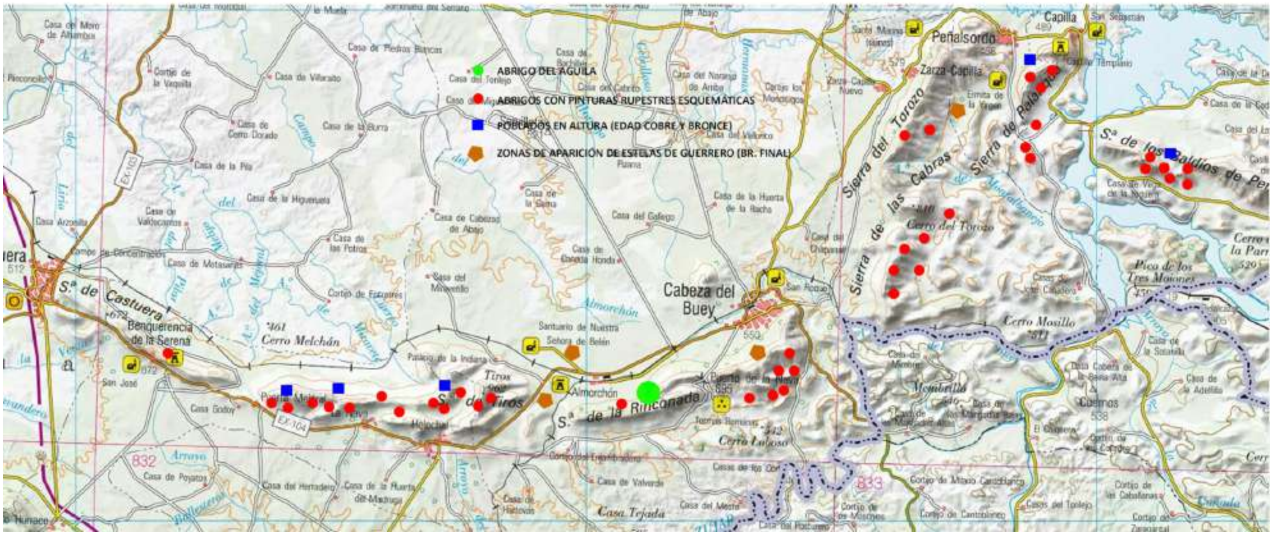
Fig. 5- Raman spectrum of sample AGU_01 (anatase).

Fig. 6 - SEM-EDS analyses on a sample of white pigment. High percentages of Ti are concentrated in the small white crystals attributable to anatase. The other elements, which were detected in the grey matrix, suggest clay minerals in the samples.

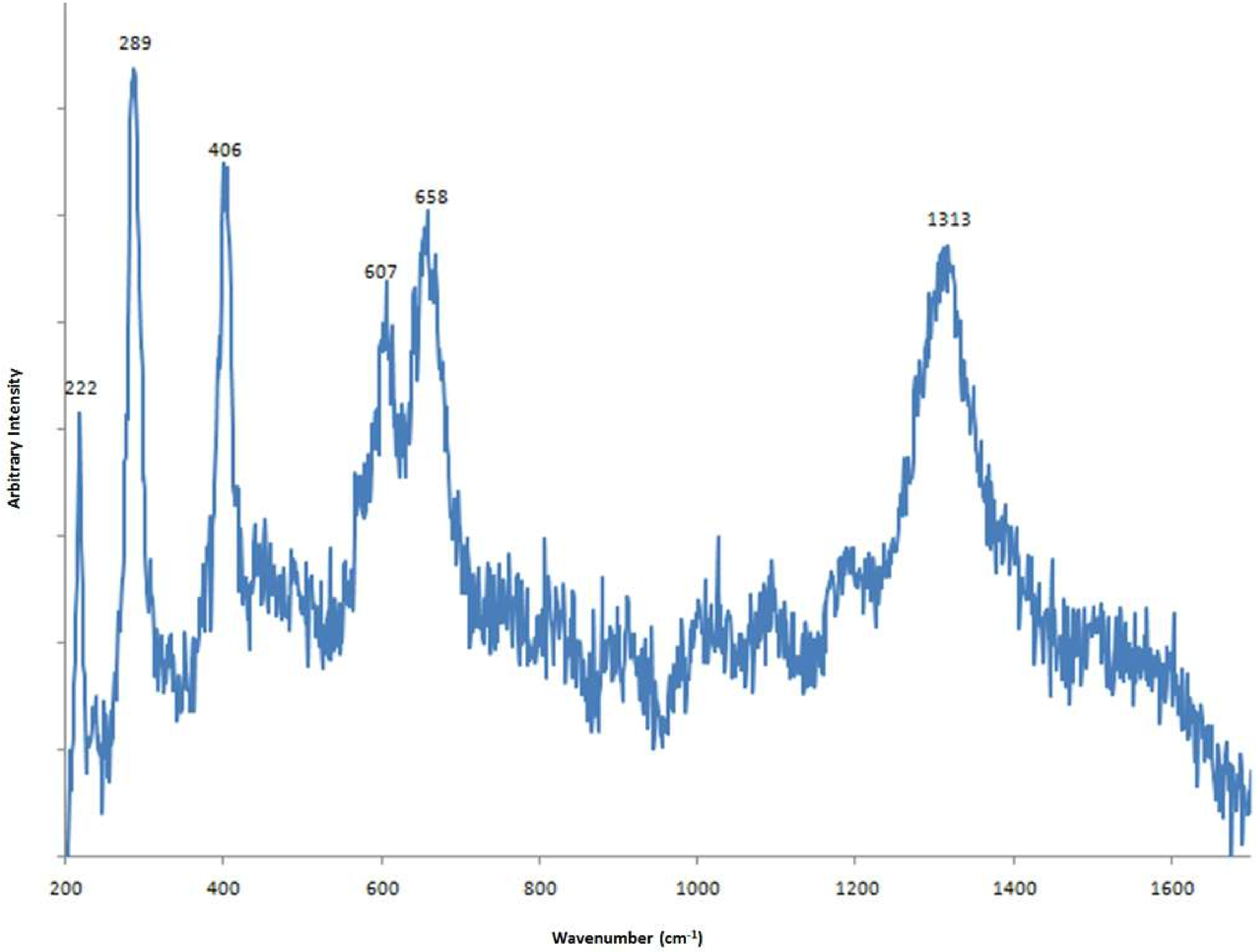
Fig. 7- Raman spectrum of sample AGU_8 (amorphous carbon)

Table 1- Results

Table 2- Semi-quantitative SEM-EDS analyses performed on two different type of red pigment (AGU_05/AGU_09)


● ABRIGO DEL ÁGUILA
● ABRIGOS CON PINTURAS RUPESTRES ESQUEMÁTICAS
■ POBLADOS EN ALTURA (EDAD COBRE Y BRONCE)
⬟ ZONAS DE APARICIÓN DE ESTELAS DE GUERRERO (BR. FINAL)
Cabeza del Buey
Peñalsordo
Capilla
Zarza-Capilla
Benquerencia de la Serena
Almorchón
S.ª de Castuera
S.ª de Tiros
S.ª de la Rinconada
Sierra del Torozo
Sierra de las Cabras
Sierra de Peñalsordo
S.ª de los Baldíos de Peñalsordo
Cerro del Torozo
Cerro Mosillo
Cerro Melchán
Cerro Loboso
Pico de los Tres Mojones


5 cm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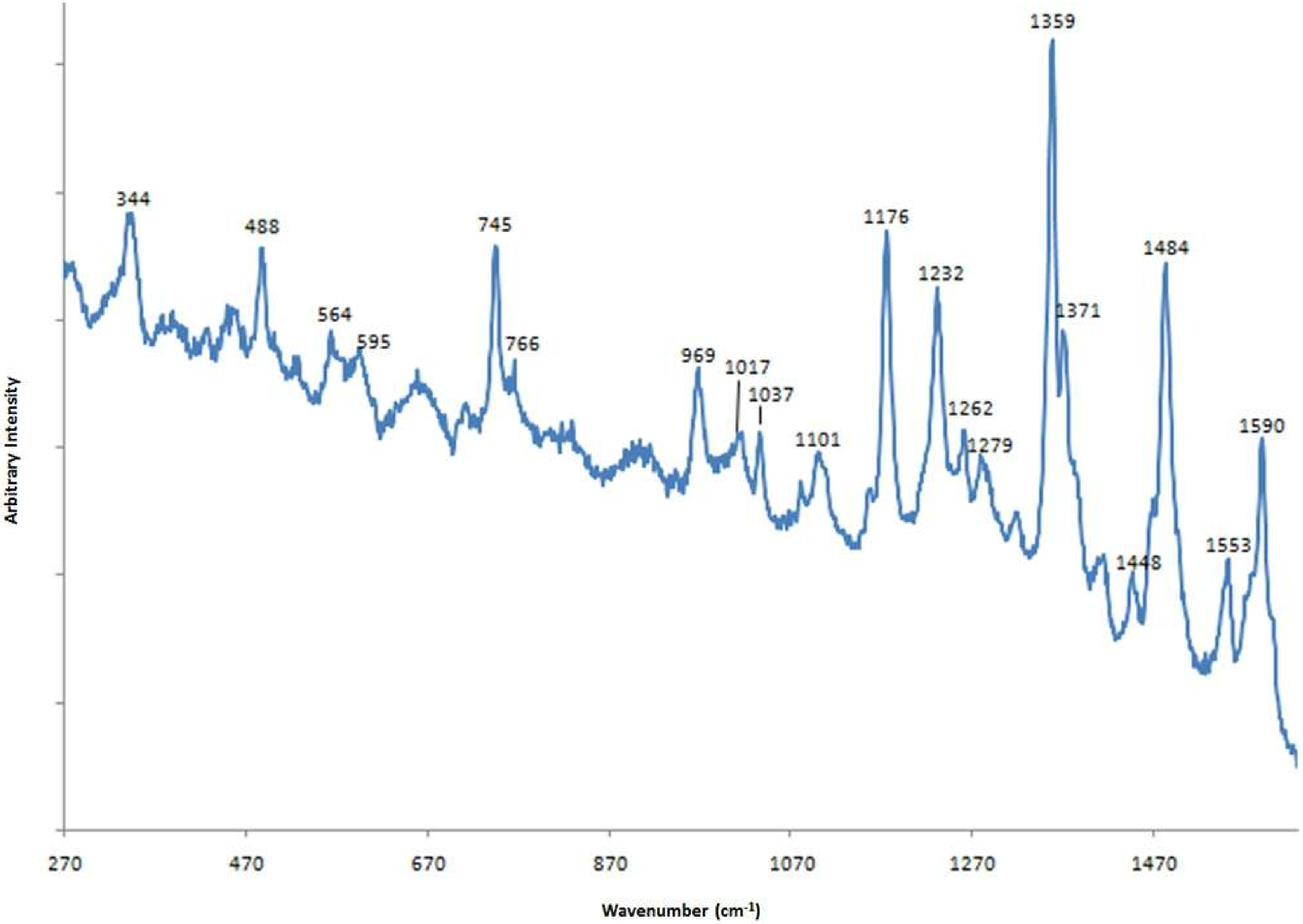
Wavenumber (cm-1)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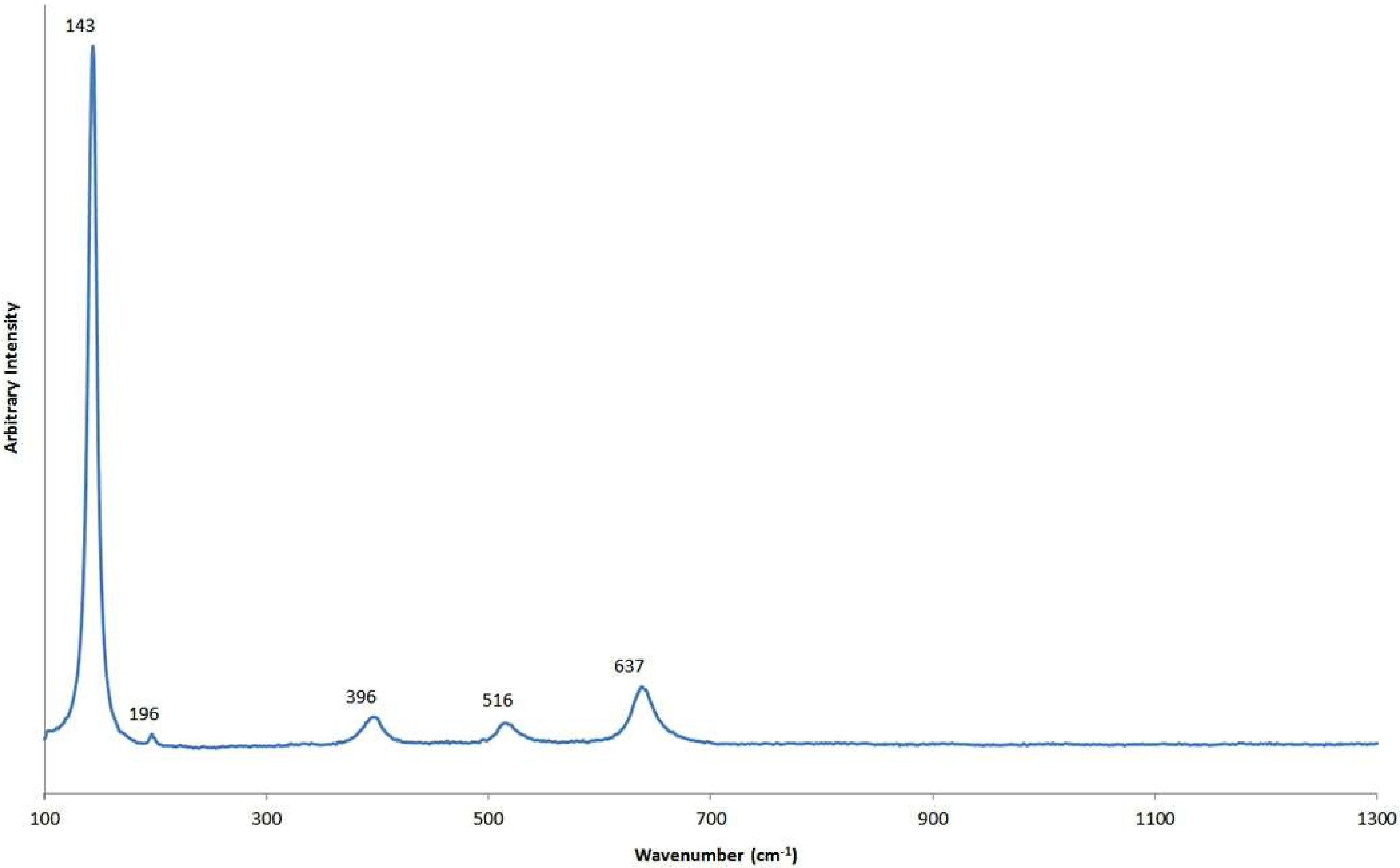
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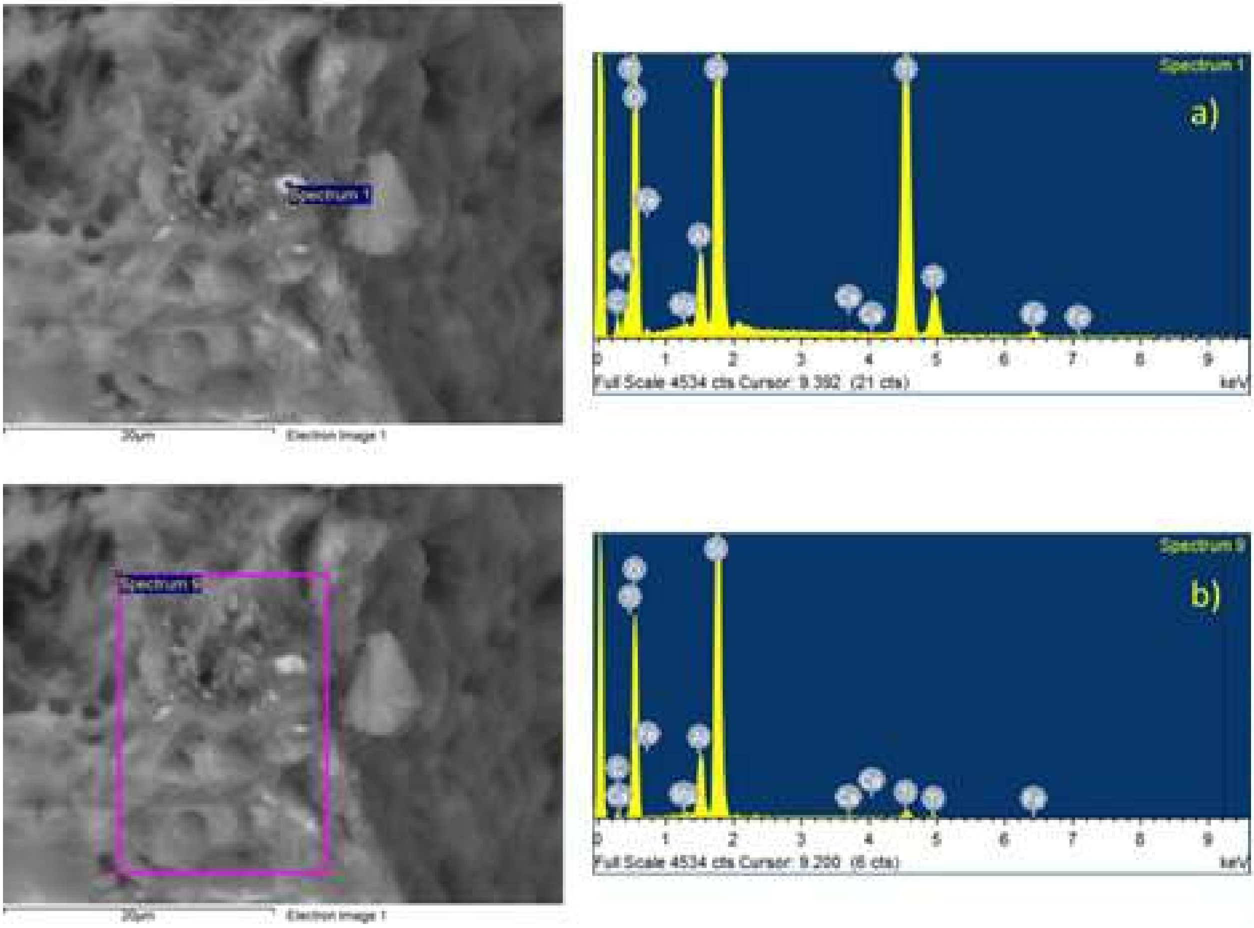
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Electron Image 1
Spectrum 1
a)
0
1
2
3
4
5
6
7
8
9
Full Scale 4534 cts Cursor: 9.392 (21 cts)
keV
Spectrum 9
20µm
Electron Image 1
Spectrum 9
b)
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1
2
3
4
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9
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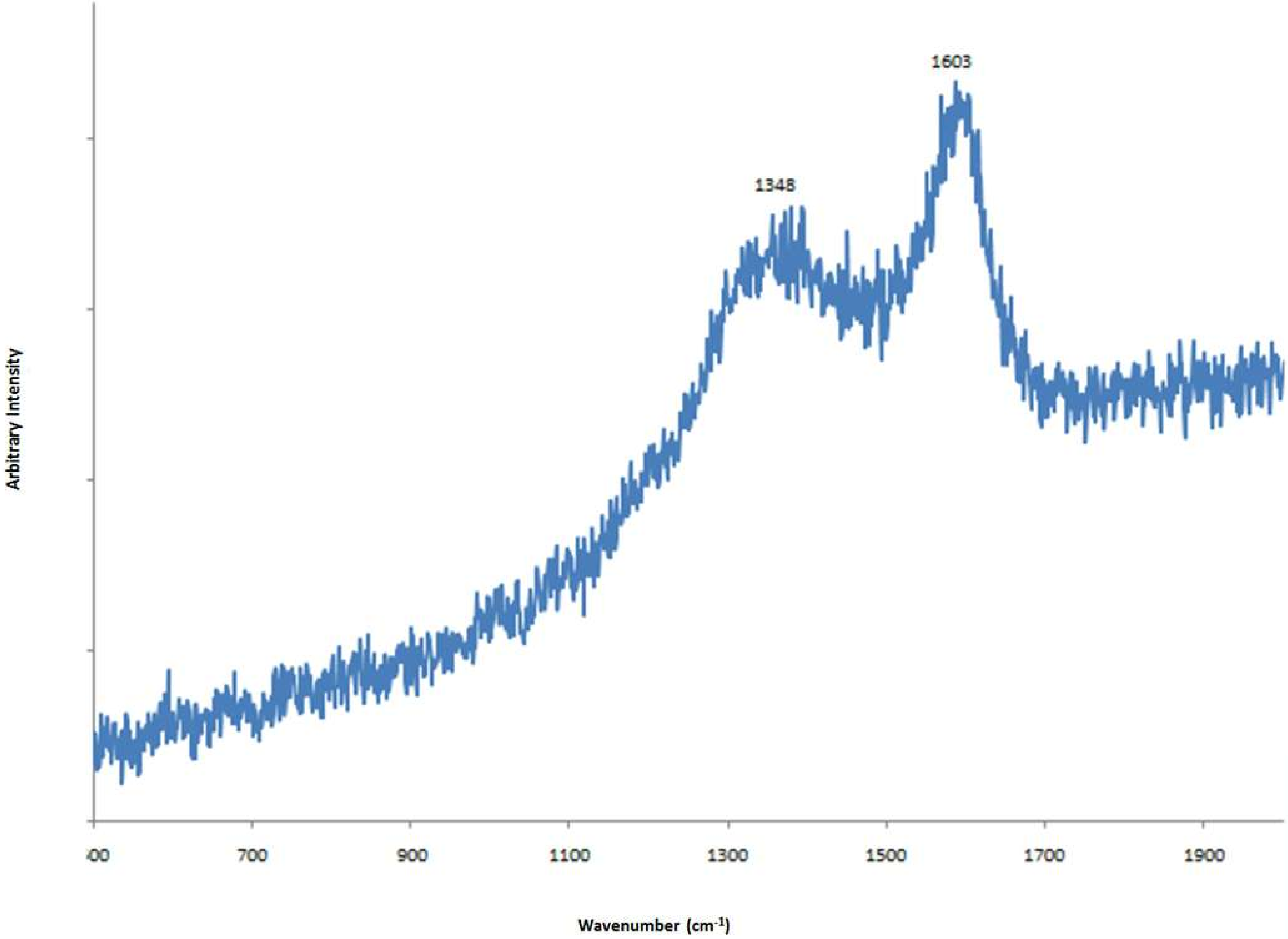
keV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Wavenumber (cm-1)