



**Experiment title:**

**Microdiffraction study of Chalcolithic rock paints from Central-West Iberian Peninsula**

**Experiment number:**

EC-726

**Beamline:**

ID18F

**Date of experiment:**

from: November 1<sup>th</sup> 2010 to: November 5<sup>th</sup> 2010

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**Shifts:**

9

**Local contact(s):**

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*Received at ESRF:*

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**Report:**

In this experiment some cave painting samples have been analyzed using micro-diffraction and micro-fluorescence techniques at the ESRF. All the paints under study belong to the so-called schematic art. The predominant colour is red and the paints are found in rocks in contact with the external atmosphere. Petrography and SEM studies permitted to identify the red pigments as iron oxides, but there is very little information on the composition and origin of post-Paleolithic pigments as well as the possible interactions between rock and painting medium. Conventional X-ray diffraction failed to identify precisely the nature of the paint, because the unavoidable presence of substrate phases.

The experiment was performed at the station ID18F, and the most suitable samples were those embedded in resin and cutted in thin sections of about 40 microns thick.

The aim of this work is to identify the crystalline phases in the pigment, substrate and possible alterations at the pigment surface and at the pigment-rock interphase. Measurements were taken separately to optimize the acquisition time and get better records.

The  $\mu$ -XRD analyses allowed to determinate some mineral phases not identified until now. Thus, the  $\mu$ -XRD supported on  $\mu$ -XRF studies, have confirmed the nature of the paintings, predominately composed by silicates and

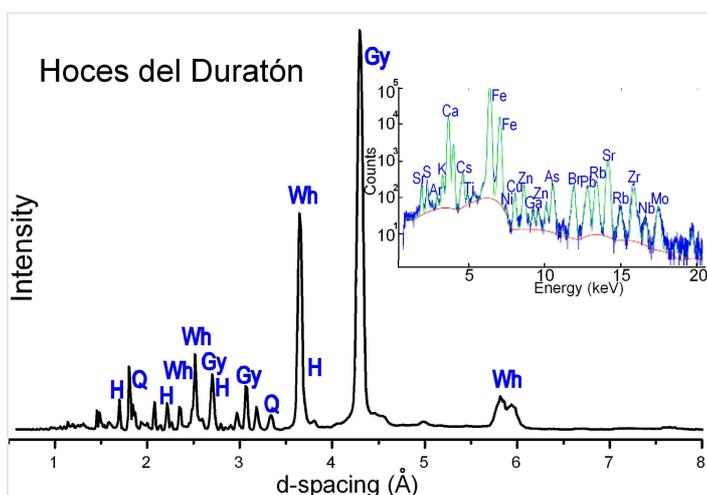


Fig. 1.- Diffractogram and fluorescence spectrum for Hoces del Duratón site, Segovia.

iron oxides in different mineral phases. The determination of these iron minerals that color the paintings, may not be the same crystalline phase.

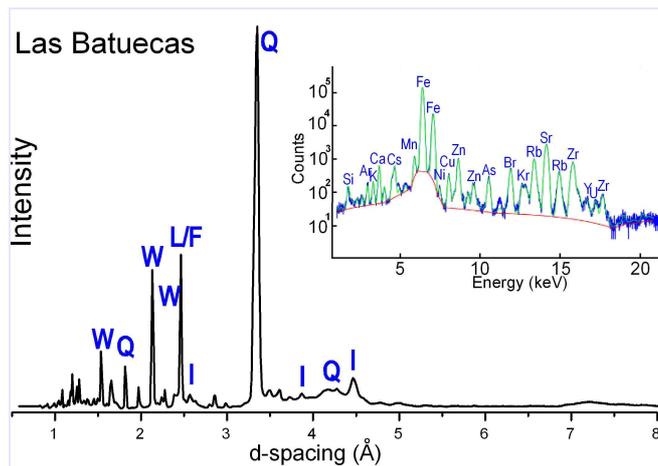


Fig. 2.- Diffraction pattern and fluorescence spectrum for Las Batuecas site, Salamanca.

The main silicate phases identified are quartz in the Hoces del Duratón site (Fig.1), with Illite/Muscovite in the Batuecas site (Fig.2); the same in Valonsadero site (Fig.3) with kaolinite as major mineral present. The silicate nature of the pigments gives an idea of the source, but a deeper study must be carried out to clarify this issue.

Regarding to iron oxides mineral phases, the identified phases are hematite and goethite in Segovia and Soria sites respectively, and lepidocrocite, ferrihydrite and wustite in Salamanca site.

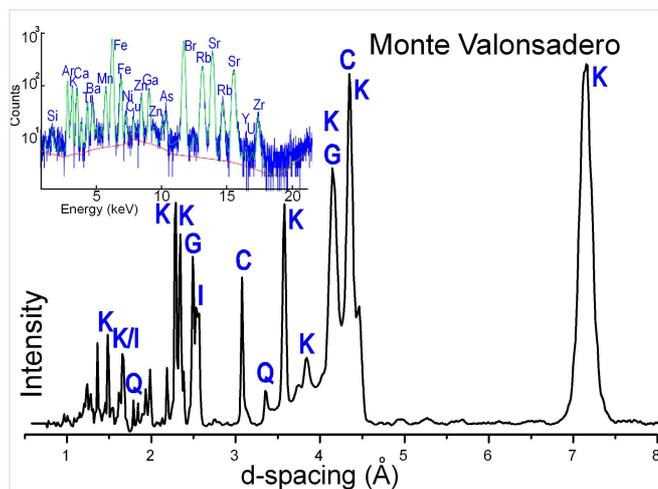


Fig. 3.- Diffraction pattern and fluorescence spectrum for Monte Valonsadero site, Soria.

Some mineral phases related to alteration processes and the influence of supporting rocks in paintings have been recognised: whewellite and gypsum in limestones substrate and monohydrocalcite in sandstone supporting rocks.

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