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Calcium oxalate AMS ^{14}C dating and chronology of post-Palaeolithic rock paintings in the Iberian Peninsula. Two dates from Abrigo de los Oculados (Henarejos, Cuenca, Spain)

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ABSTRACT

Since 2005 we have been utilizing accelerator mass spectrometry (AMS) ^{14}C dating in research on calcium oxalate crusts associated with open air rock art of the Iberian Peninsula. In this paper we present two dates linked with three eye-idol pictographs at Abrigo de los Oculados (Henarejos, Cuenca, Spain). Radiocarbon ages for these motifs agree with the expected iconography-based archaeological chronology. Such oxalate dates could provide an independent basis for evaluating chronological theories for post-Palaeolithic sites, designated in the UNESCO World Heritage List as Rock Art of the Mediterranean Basin on the Iberian Peninsula.

1. Introduction

The chronology of open air rock paintings in the Iberian Peninsula is still subject to scientific debate due to a paucity of direct radiocarbon dates. Rock paintings in this region include at least three different styles of art: Levantine, Schematic, and Macroschematic (Fig. 1). These traditions, especially Levantine art, possess an iconographic richness useful in elucidating social structures, hierarchies and conflicts, technologies, beliefs and symbolism of these prehistoric societies. These styles become more significant given that they are possibly related to the first appearance of herder and farming economies in Western Europe. However, their heuristic utility remains uncertain due to lack of a solid chronological frame of reference.

Calcium oxalate coatings have been identified in many rock art panels. In previous papers (Ruiz et al., 2006, 2009) we have proposed radiocarbon dating of these coatings offers a feasible method for testing chronological models based on stylistic comparisons with mobiliary objects. Radiocarbon dating of oxalate accretions may also help establish chronological limits for pictographs that, for whatever reason, cannot otherwise be dated directly. Here we extend this research, presenting *post quem* and *ante quem* radiocarbon dates for painted 'eye-idols' at Abrigo de los Oculados (Henarejos), a rock art site in the Sierra de las Cuerdas group (Cuenca, Spain). We used AMS ^{14}C to analyze samples from oxalate coatings underlying, and also overlying pictographs there. Chronology of the eye-idol motif has been inferred by archaeological methods, which could afford an independent way to refute or confirm the radiocarbon age bracket of calcium oxalate dates.

Current chronology of post-Palaeolithic rock paintings of the Iberian Peninsula is based mainly on stylistic comparisons with mobiliary items. Schematic and Levantine art have for decades been considered mutually interdependent in unilineal evolutionary schemes (Ripoll, 1964, 2001; Beltrán, 1968). Rock art described as Macroschematic was incorporated later into the same debate (Hernández et al., 1988).

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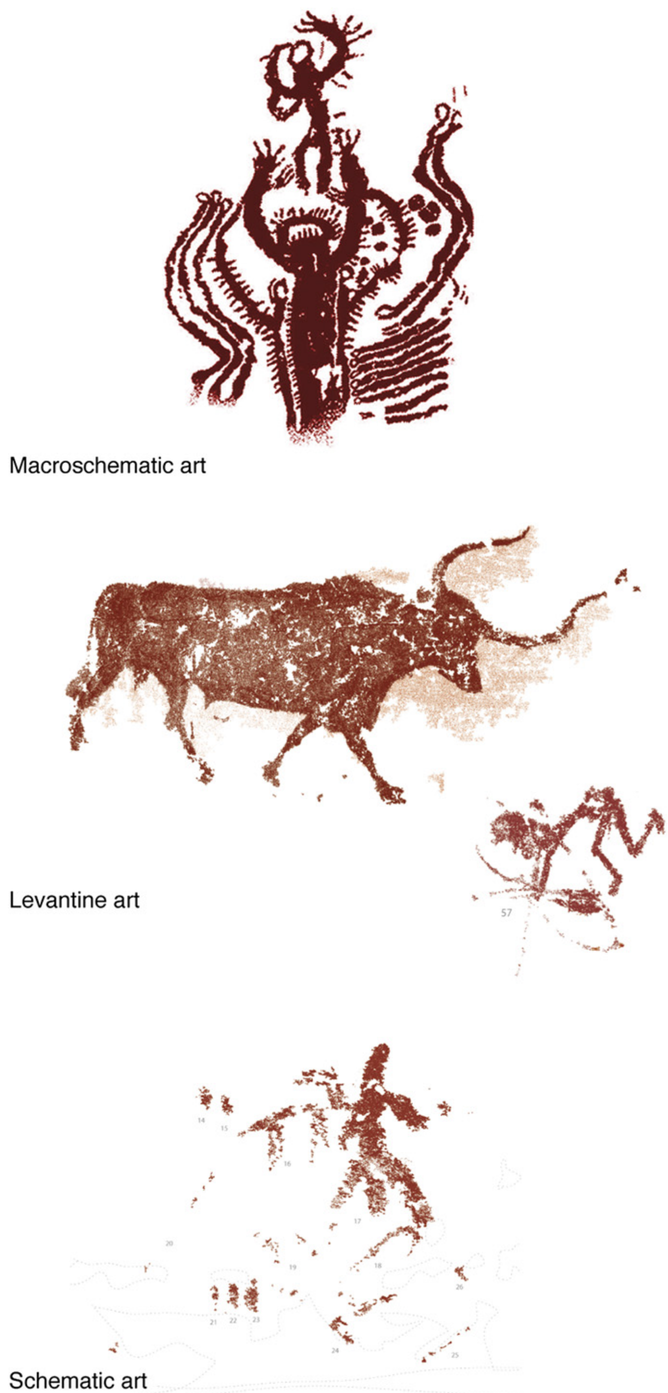

Fig. 1. The styles of Iberian post-Palaeolithic rock paintings: Macroschematic (Barranc de l'Infern IV, Beniarrés, Alicante) (Martí and Hernández, 1988), Levantine (bull: Selva Pascuala, Villar del Humo, Cuenca, and archer: Cueva del Tío Modesto, Henarejos, Cuenca) (Ruiz, 2006a,b) and Schematic (Peña del Castellar, Villar del Humo, Cuenca) (Ruiz, 2006b). Tracings not to scale.

Schematic iconography is characterized by anthropomorphs and zoomorphs reduced to simplified shapes, rendered mainly with broad strokes forming irregular lines. This style also features abstract motifs such as dashes and dots, and a large number of idol shapes (Breuil, 1933; Acosta, 1968), including eye-idols. Schematic art is distributed across the whole Iberian Peninsula, and is suggested to date from the Neolithic to Chalcolithic eras, based on many parallels with pottery and mobiliary items such as idols or steles (Hernández et al., 1988; Torregrosa and Galiana, 2001). Currently there is an absolute consensus on this chronological frame, but questions arise. This style has been defined mainly by contraposition to Levantine art, which includes naturalistic animals and stylized humans, often figuring in complex narrative scenes (Beltrán, 1982). Consequently, every non-naturalistic motif in the Prehistory of Iberian Peninsula, from Neolithic to the Iron Age, has been included under this broad concept of Schematic art. Some researchers are now beginning to describe distinct styles within Schematic art (Sanchidrián, 2001), such as so-called 'black Schematic Cave art' (Sanchidrián et al., 2001; García et al., 2005); Ancient Schematic art (Hernández, 2006) which is composed of zigzags, and is currently thought to have resulted from the spread of Macroschematic art, or Megalithic art (Bueno et al., 2007), defined by many iconographic coincidences with it.

After the discovery of Levantine art in 1903, Schematic art became integrated as a stage in unilineal evolutionary schemes of the time. If Levantine art originated in the Palaeolithic (Breuil, 1920; Breuil and Cabré, 1909; Obermaier, 1924), and it was thought it always underlaid Schematic, the latter should be Neolithic (Breuil, 1933; Cabré, 1915; Hernández-Pacheco, 1924). By the mid 20th Century both styles were considered post-Palaeolithic, and Schematic art was interpreted as a logical consequence of evolution from stylized Levantine figures, as influenced by Neolithic and Metal Age Mediterranean cultures (Ripoll, 1964) and their iconography (Breuil, 1933; Acosta, 1968).

Evidence more recently discovered has begun to complicate this simple sequence. Beltrán (1968), whose evolutionary model for Levantine art did not include Schematic art, saw some Schematic figures underneath Levantine ones at La Sarga (Alcoy, Alicante). Soon after, a similar order of superimpositions was indicated for other sites in Eastern Iberia (Fortea, 1974, 1975). The discovery of Macroschematic art in Alicante province in the 1980s decisively altered the previous chronological model, because it underlay Levantine figures at several sites (Hernández et al., 1988).

The chronology of Macroschematic art has been established by stylistic parallels with decorations on cardial pottery from the Early Neolithic of Cova de l'Or (Beniarrés, Alicante) and other archaeological sites (Martí and Hernández, 1988; Martí and Juan-Cabanilles, 2002). The iconography of this style includes schematic anthropomorphs and multiple snake-like figures, sometimes larger than 1 m. Based on these parallels, it was proposed that Macroschematic art falls between 5460 and 5230 cal BC (Fairén, 2004). For these investigators this new style is older than Levantine and Schematic art in the Valencian region. They have also proposed some stylistic parallels for Levantine figures in epicardial pottery from these sites (Martí and Hernández, 1988; Martí and Juan-Cabanilles, 2002). Thus, recent years have seen a growing tendency to place the origin of these three styles in the Neolithic, but with significant differences as to their role in the neolithisation process of the Iberian Peninsula (Hernández and Martí, 2000-2001; Hernández, 2006; Cruz and Vicent, 2007).

The chronology of Levantine art is still open to debate however, as other researchers have questioned these analyses on various points. Some have rejected the pottery decoration parallels, considering them to be inadequate (Baldellou, 1988; Mateo, 2002; Viñas et al., 2010). Others consider that some of the superimpositions of Levantine pictographs over Macroschematic ones are incorrect (Ros, 2011). Likewise, the discovery of Levantine style engravings (Utrilla and Villaverde, 2004) has reopened the old debate on the continuity between Palaeolithic and Levantine art (Viñas et al., 2010).

This complex panorama demands scientific dating to clarify. Here we present results of AMS ^{14}C dating for two calcium oxalate samples collected in Abrigo de los Oculados, both associated with typical Schematic pictographs. These results allow us to examine the feasibility of applying oxalate dates to rock art images, as a possible contribution to the establishment of a radiocarbon frame of reference for post-Palaeolithic pictorial styles of the Iberian Peninsula.

2. Abrigo de los Oculados

The two samples of calcium oxalate crust we dated from Abrigo de los Oculados (Fig. 2) were collected from the places indicated in Fig. 3. This small shelter of reddish Triassic sandstone is barely covered by a small overhanging shelf (Ruiz, 2006a). Twenty-nine pictographs of Schematic style are preserved on this panel. Among these, three stand out because their formal features match the usual typology of eye-idols (Pascual, 2010) found on mobiliary items (Almagro, 1973), and in Schematic rock art. Two eyes, sometimes with radiating lines, form the central structure of this motif, highlighted by bent or curved lines above that can be interpreted as eyebrows. This structure is complemented with pairs of parallel curved lines below the eyes, usually referred to as a 'face tattoo' (Siret, 1908). At this site, the eye-idol structure is juxtaposed with a basic sketch of an anthropomorph.

The three eye-idol pictographs are surrounded by other Schematic figures of less defined typology, painted in reddish shades. This color could have resulted from use of red ochre, i.e., clay with a high concentration of iron oxides. *In situ* Raman analyses of the pigment, performed with a portable Raman spectrometer (*innoRam-785H, B&W TEK, Inc.* Newark, USA) showed significant levels of fluorescence, which often results from presence of clay minerals of the phyllosilicate group. This hypothesis will need to be verified through other chemical analysis techniques such as XRD and FTIR.

Fig. 2. Photograph of the Abrigo de los Oculados.

The entire panel at Abrigo de los Oculados has a pale ochre orangish shade, 5 YR 7/2 (Munsell Rock-Color, 1995), due to oxalate patination, with some areas covered by a lighter beige color, 10 YR 8/2 (Munsell Rock-Color, 1995). The lighter crust covers the entire leftward side of the panel, plus some portions on the right. The effects of this patination can be seen over eye-idol no. 10, the left part of which is faded (Fig. 4). The chemical composition and microlamination of these coatings has been studied by thin section of a microsample, collected in 2005. Direct observations by stereoscopic microscope *Nikon SMZ*, supported by macrophotographs, confirm that the light beige crust overlies the pictographs, while the pale ochre orangish one is underneath them (Fig. 4). We consider that these two coatings originated in different periods, that their radiocarbon ages should be consecutive, and bracket the age of the pictographs situated between them.

3. Rock art dating methods

Despite significant efforts in recent decades, determining the chronology of open air rock art remains one of the greater challenges for rock art studies entering the 21st century (Bednarik, 2002; Rowe, 2005, 2009; Watchman et al., 2005). In contrast to deep cave art, open air paintings and engravings generally lack well-defined archaeological contexts as foundations for unequivocal cultural and chronological attribution. For example, the archaeological contexts for rock art sites in the Sahara (Denyer et al., 2007) and in Baja California (Magar and Dávila, 2004; Petit and Rubio, 2006) have not allowed for clear chronological sequences to be established. These rock art ensembles are in locations that were utilized over millenia by people of different cultural traditions. More recent cultures have reused these sites, or even appropriated the rock art and altered it over time. But usually, the main obstacle for defining reliable chronological contexts is the lack of archaeological records that can be associated with specific prehistoric rock art.

Additional difficulties exist for obtaining absolute dates for open air rock art (Bednarik, 2002). One well-known problem is that paint preparations used must include some organic material to contain carbon for dating. If organic matter was present in the paint originally, it is further necessary that any organic compounds persist as such to the present time without exchanging carbon. Furthermore, even if paints used contain organic substances, ethical considerations based on the size of samples needed to carry out AMS ^{14}C dating may be prohibitive. One notable exception to this is when charcoal was used as pigment. In spite of all this, pigments and binders from numerous sites around the world have been dated by AMS ^{14}C over the last two decades (for example: Cole and Watchman, 2005; Fullola et al., 1994; Mori et al., 2006; Nelson et al., 1995; Petit and Rubio, 2006; Rowe, 2005, 2009; Russ et al., 1990; Valladas, 2003; Valladas et al., 2001; van der Merwe et al., 1987; Watchman and Cole, 1993; Watchman et al., 2002) to list only a few references. Rowe (2012) published a bibliography of references for rock art thus dated, as complete as he was able to compile. It includes many hundreds of publications.

Rock paintings are often associated with organic and inorganic coatings that form on rock surfaces. Based on their stratigraphic relationship with rock art, these accretions have been used as an alternative to pigments for radiocarbon dating, requiring no organic material be present in the paintings (Cole and Watchman,

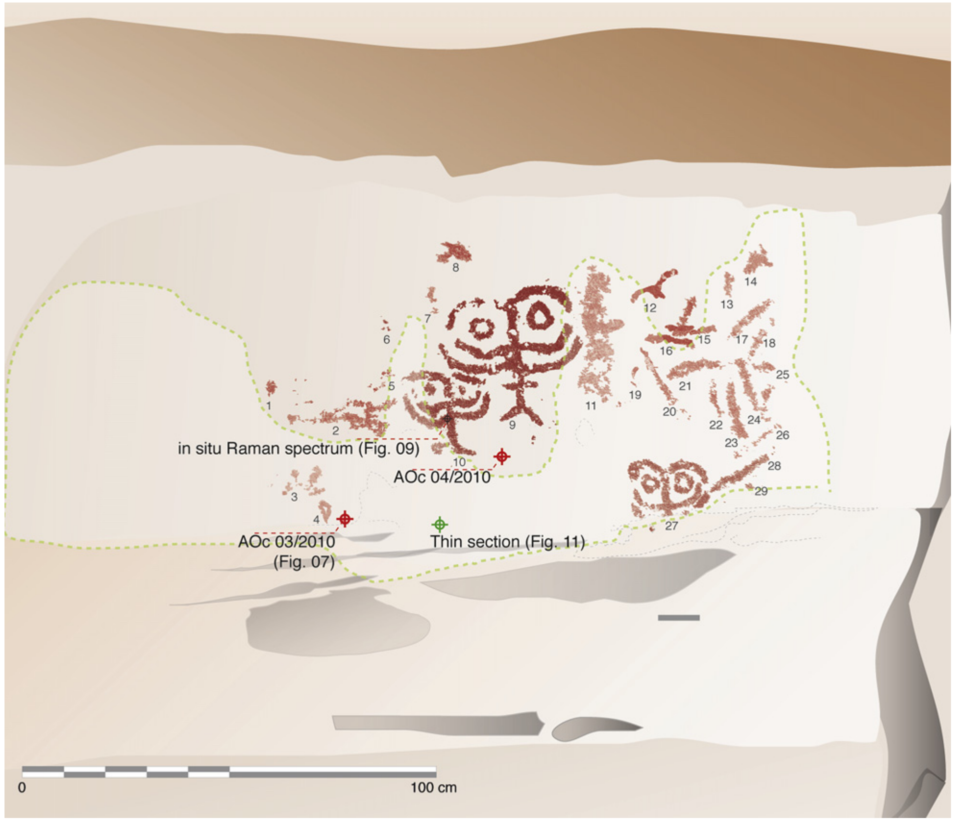

Fig. 3. Location of the samples collected for AMS ^{14}C in Abrigo de los Oculados panel (red marks), sampling point for the thin layer, and extension of the accretionary crust overlying paintings (dotted area of lighter color). It is indicated also the position of one of the points analysed *in situ* with μ-Raman. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

2005; Mazel and Watchman, 1997; Steelman et al., 2002; Rowe and Steelman, 2003; Ruiz et al., 2006). Steelman et al. (2002) and Rowe and Steelman (2003) reported dates on both an oxalate-containing calcite crust over a painting, as well as the painting itself. As expected, the oxalate crust was roughly half the age of the pictograph. That correspondence lends confidence to the oxalate technique. This approach can also afford a chronological context for pictographs of small size, which would be seriously damaged by removal of pigment samples. Levantine art is a good example of this situation because the average size of the figures is 10–30 cm, and charcoal has rarely been reported (Ballester et al., 2010; Montes and Cabrera, 1991; Ruiz et al., in press).

Fig. 4. A light beige accretionary crust overlies the left part of the eye-idols in the middle of the panel. At the same time, they were painted over a pale ochre orangish coating. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Radiocarbon dating of calcium oxalate (CaC_2O_4) crusts or biofilms was first used as a way to establish minimum/maximum ages for rock art in Australia in the early 1990s (Watchman, 1991). Since then calcium oxalate has been successfully dated in that country (Watchman and Campbell, 1996), in South Africa (Mazel and Watchman, 1997, 2003), the southwestern USA (Russ et al., 1995, 2000) and Brazil (Steelman et al., 2002; Rowe and Steelman, 2003). This carbon-bearing mineral has also been dated in Argentina (Hedges et al., 1998) and Mexico (Watchman et al., 2002). In both latter cases, the presence of oxalate was interpreted as a decomposition product of a binder present in the paints used, derived from cacti.

There are several hypotheses on the origin of calcium oxalate coatings, ranging from results of human activity (Rampazzi, 2004), to metabolic action of lichens, bacteria and microbes inhabiting the outer layers of rock faces (Beazley et al., 2002; Hess et al., 2007; Krumbein et al., 2003). It has also been proposed that pictographs may sometimes abet the formation of these crusts, due to bacteria and fungi feeding on chemical components of the paint (Cole and Watchman, 2005). For our part, we consider metabolic activity of lichens or microorganisms offers the more likely explanation for the origin of calcium oxalate crusts (Hernanz et al., 2008).

Whewellite is one of the hydrated forms of calcium oxalate, a salt of oxalic acid. The IUPAC (International Union of Pure and Applied Chemistry) name of oxalic acid is 'ethanedioic acid'. The crystal structure of whewellite is built with oxalate anions, calcium cations and water molecules. The oxalate anion, the base of oxalic acid, and the acid itself, contains two carbon atoms linked with a "sigma" bond formed from sp3 hybrid orbitals from each carbon

Fig. 5. Location of the specimens collected for AMS ^{14}C in Selva Pascuala, panel 1, and a nearby shelter without rock art.

atom, a typical molecular structure of organic compounds. Moreover, two carboxylic groups constitute the oxalate anion and oxalic acid, the typical functional group of organic acids. Calcium oxalate is practically insoluble in water; it is inert and very stable under UV radiation. Calcium oxalate may be preserved on rock surfaces during thousands years without alteration.

Our research team began dating oxalate crusts in Spain in 2005. This line of investigation was incorporated into several research projects, funded and approved by the Spanish cultural authorities. Our studies sought to analyze the chemical composition and chronology of Levantine and Schematic paintings (Hernanz et al., 2006, 2010). The initial results were radiocarbon dates from Cueva del Tío Modesto (Henarejos, Cuenca), for calcium oxalate crusts associated with Levantine and zigzag pictographs there (Ruiz et al., 2006). This was followed by new radiocarbon dates for nearby shelters that also produced encouraging results (Ruiz et al., in press). Together, these are the only radiocarbon dates published for an organic substance in known relation with post-Palaeolithic open air rock paintings in the Iberian Peninsula.

4. Experimental procedure

Two samples from Abrigo de los Oculados (Fig. 3), and two others from Selva Pascuala (Villar del Humo, Cuenca, Spain) (Fig. 5), were collected in mid-2010. They were obtained and handled wearing disposable latex gloves and hygienic masks, using sterile surgical blades. A new surgical blade was utilized for each. Small crevices and partially detached flakes were used to facilitate sample removal, minimizing the risks of damage to the rock surface of the panel. Magnifying glasses (4×) were used for this purpose. Sample locations were carefully selected to ensure that no paint was removed along with the oxalate crusts, as we were not authorized to collect pictograph samples. Macrophotographs of the sampled areas were taken before and after removal. Samples were wrapped in aluminum foil, and placed in hermetically sealed plastic bags (Rowe, 2001; Hernanz et al., 2010).

Our selection of locations for sampling at Abrigo de los Oculados and Selva Pascuala were based on the stratigraphic position of the oxalate coatings. At Abrigo de los Oculados, we tried to collect one sample of the crust covering the pictographs and another of the

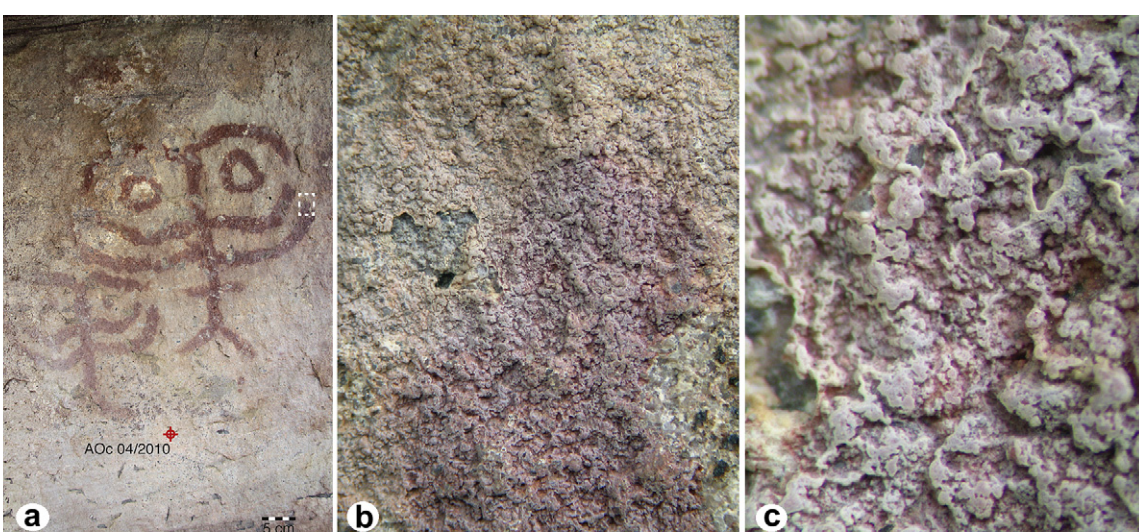

Fig. 6. Sample AOc 03/2010 was removed from a point that the overlying crust is fading pictographs. The appearance of this coating is similar to that covering some parts of the central eye-idols figures. In picture (a) it is shown the point enlarged in (b) and (c); b- microphotograph at 5× of the end of one of the face tattoos, an area which is less faded that point AOc 03/2010 or left part of the eye-idol no. 10; c- microphotograph at 25× of the same area (see Fig. 8b and c for comparison).

Fig. 7. Place where specimen AOc 03/2010 was removed. Picture taken after collection. The tip of the surgical blade is placed directly below the point where the flake was removed. Rock base was not affected by removal of this sample.

underlying layer, with the aim of getting minimum and maximum ages for the painting event, thus constraining its age. The appearance of the coatings at these two sampling points was very similar to that of the coatings in stratigraphic relationship with the eye-idol pictographs nearby (Fig. 6). Sample AOc 03/2010, measuring 0.3 × 0.9 cm, was collected from the lighter shade crust covering the pictographs (Fig. 7). Sample AOc 04/2010, with a size of 0.4 × 0.6 cm, was removed from the underlying accretionary film, in an area without the oxalate crust that is fading in other parts of the frieze (Fig. 8).

Special care was taken in the collection of sample AOc 03/2010 to avoid contamination of the coating. Although the oxalate crust underlying the paintings seems to cover the entire panel, the area from which this specimen was collected displays a noticeable textural change, probably because the coating was eroded before it became covered again with oxalate. We cannot ascertain this, however, because the sample we obtained was too small for thin section and AMS ^{14}C dating. We collected it from a flake loosened by microerosion of the panel, attempting to sample the upper layer only. However, we cannot confirm complete absence of any underlying crust in the sample.

Table 1
Oxalate samples chemically pretreated at EMU.

Sample ID	Mass, mg	Description
SP 01/2010	626.4	Brown material
SP 02/2010	973.4	Reddish-pink material
AOc 03/2010	146.1	Reddish-pink material
AOc 04/2010	104.3	Reddish-pink material

The same procedure was used to remove two additional samples from Selva Pascuala shelter (Fig. 5). In Selva Pascuala panel 1 (SP 01/2010), one specimen was collected from the crust apparently covering the large Levantine bull in this panel. Another was gathered at a shelter 20 m away from the rock art site (SP 02/2010), to serve as a control sample for SP 01/2010, and for a previous published dating from Selva Pascuala panel 2 (Ruiz et al., 2009).

The four oxalate samples were analysed by Raman microscopy with a confocal microscope (*Jobin Yvon LabRam-IR HR-800*) in the Departamento de Ciencias y Técnicas Fisicoquímicas of UNED (Madrid), following procedures described elsewhere (Hernanz et al., 2006, 2008). Further *in situ* surface analyses of the Abrigo de los Oculados panel were carried out using a portable μ-Raman *innoRam-785H*, with laser excitation at 785 nm, a Peltier cooled CCD detector, and a fiber optic microprobe at 10× magnification. The results of these *in situ* analyses have corroborated the former laboratory findings.

The identification of calcium oxalate by Raman spectroscopy enabled us to plan AMS ^{14}C dating. Our four oxalate specimens were sent to Eastern Michigan University (EMU) for chemical pretreatment. All glassware, filters, and aluminum foil were baked overnight at 500 °C in a muffle furnace to remove all organic traces prior to use. Each sample was crushed in a clean mortar and pestle, weighed, and transferred to a cleaned microcentrifuge tube for treatment. Table 1 shows the masses of each sample and a description of their appearance. One mL of 1 M phosphoric acid was added to each sample. Bubbles were observed in sample 02/2010:SP, indicating the presence of carbonates. The acid-treated samples were sonicated at room temperature for 30 min. Following sonication, samples were centrifuged for one minute, and the solution removed with a clean Pasteur pipet. Samples 01/2010:SP and 02/2010:SP were treated a second time with acid to ensure removal of carbonates; no further decomposition of carbonates was

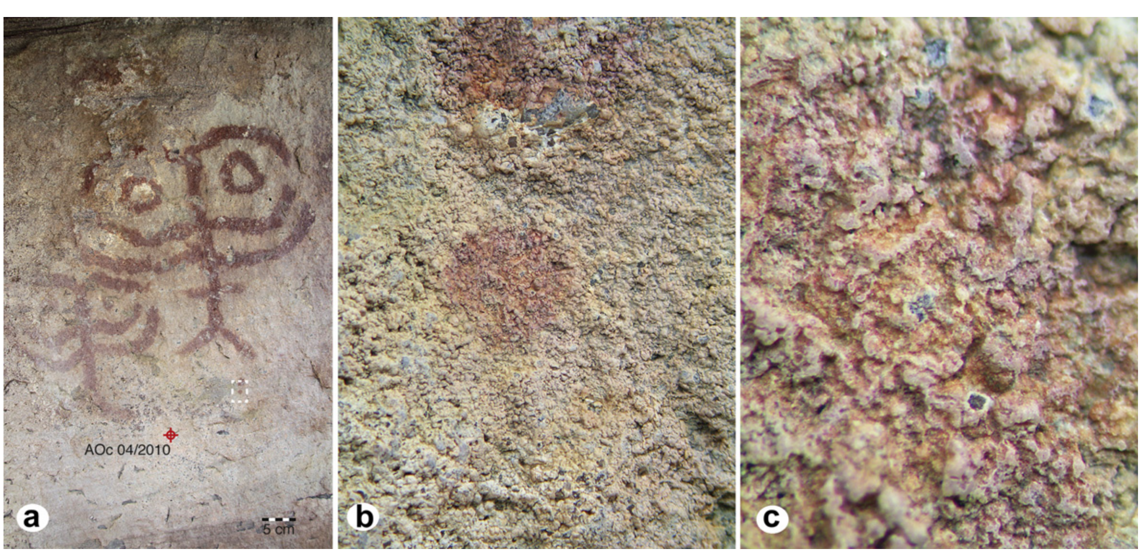

Fig. 8. Sample AOc 04/2010 was collected in an area that overlying crust is not fading pictographs. In picture (a) it is shown the point sampled and the area enlarged in (b) and (c); b- microphotograph at 5× of a tiny painting dot, which is not faded by the crust; c- microphotograph at 25× of the same dot (see Fig. 6b and c for comparison).

observed. Universal pH paper indicated that the solutions had a pH < 2 after treatment.

The oxalate solids were rinsed with deionized water (18 MΩ, Barnstead NANOpure), then treated with pH 8 phosphate buffer (1 M in phosphate ion) to remove humic acids. This procedure was developed by the Armitage laboratory as a less damaging way of removing humics from fragile artifacts, and yields radiocarbon dates indistinguishable from those for which the more caustic sodium hydroxide was used (Ellis, 2008; Li, 2010). After sonication in the phosphate buffer for 30 min at room temperature, the solutions were clear, indicating humics were removed.

The samples were rinsed with deionized water, followed by 1 M phosphoric acid, and filtered using borosilicate glass binder-free filters. The dry material on the filter was wrapped in clean aluminum foil, then labeled and repackaged for submission to the Center for Accelerator Mass Spectrometry at Lawrence Livermore National Laboratory (CAMS-LLNL) for AMS radiocarbon analysis.

One microsample of the bedrock sandstone, previously collected at Abrigo de los Oculados in 2005, was thin-sectioned in order to study its microstratigraphy. The thin section was prepared in the Laboratorio de Preparación de Muestras del Departamento de Mineralogía y Petrología of the Universidad de Granada (Spain). The study utilized a petrographic microscope with polarized light, and was carried out in the facilities of the Instituto Gemológico Español (Madrid, Spain) and the Departamento de Ciencias y Técnicas Fisicoquímicas of UNED (Madrid). Additionally, the thin section was analysed by Raman spectroscopy.

5. Results and discussion

Calcium oxalate was identified in the laboratory by Raman spectra as whewellite ($CaC_2O_4 \cdot H_2O$). This carbon-bearing mineral displays characteristic Raman bands at 1464/1492, 1629 and 896 cm^{-1}. A small quantity of gypsum was also present. Similar results were obtained by *in situ* Raman analyses at Abrigo de los Oculados (Fig. 9).

AMS ^{14}C dating was conducted at the CAMS-LLNL facility. After graphitization there, the two samples from Selva Pascuala produced less than 20 µg of carbon, so AMS ^{14}C was not attempted. The specimens from Abrigo de los Oculados were dated successfully. Moreover, no carbonates were detected in either of these two samples, so the reservoir effect should be ruled out. The resulting radiocarbon ages were calibrated using *OxCal* 4.1 software (Bronk Ramsey et al., 2009; Reimer et al., 2009) (Fig. 10). The results are shown in Table 2.

Two calcium oxalate coatings were observed in the thin section (Fig. 11), separated by a discontinuity band. These two layers are probably the result of two successive colonization events by lichens or microorganisms, separated by a time lapse. Our findings from thin section study agree with previous observations of the two superimposed calcium oxalate crusts on portions of the wall of this shelter.

The main objective of this work was to examine the reliability of AMS ^{14}C dating of calcium oxalate crusts for comparison with pictographs with an age considered reliable based on archaeological inference. The radiocarbon ages for both samples from Abrigo de los Oculados suggest that the three eye-idols there were painted in a temporal frame between 3630–3365 cal BC (2σ) and 910–540 cal BC (2σ). These dates fall between the middle of the 4th millennium cal BC and beginning of the 1st millennium cal BC, a range of dates that agrees with the expected chronology for this iconographic motif in the Iberian Peninsula. The younger limit is very close to the radiocarbon age of a sample collected from a nearby shelter some years ago (Ruiz et al., 2006). That sample, TMD-1 (see. Table 4), was removed from an area of the panel where

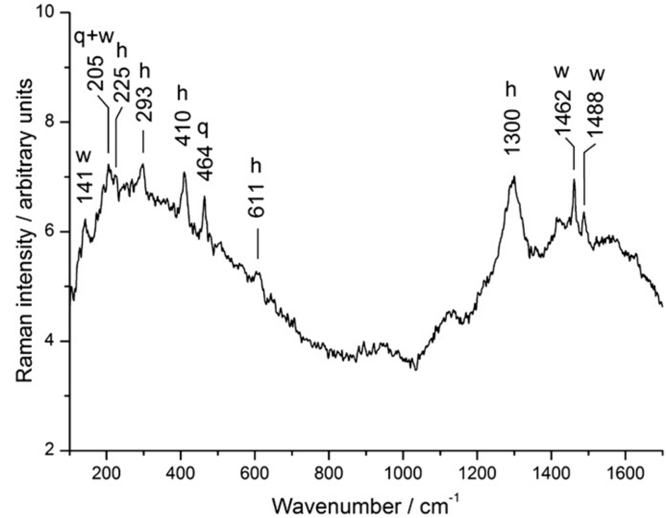

Fig. 9. Raman spectrum of the point 005 of the painting panel in Abrigo de los Oculados obtained *in situ*. Labels: h, haematite; w, whewellite; q, α-quartz.

a figure was partially broken by flaking of the wall. The area was covered later with a calcium oxalate crust. This similarity could indicate that sample AOc 03/2010 was, at least partially, free of contamination from the underlying accretion.

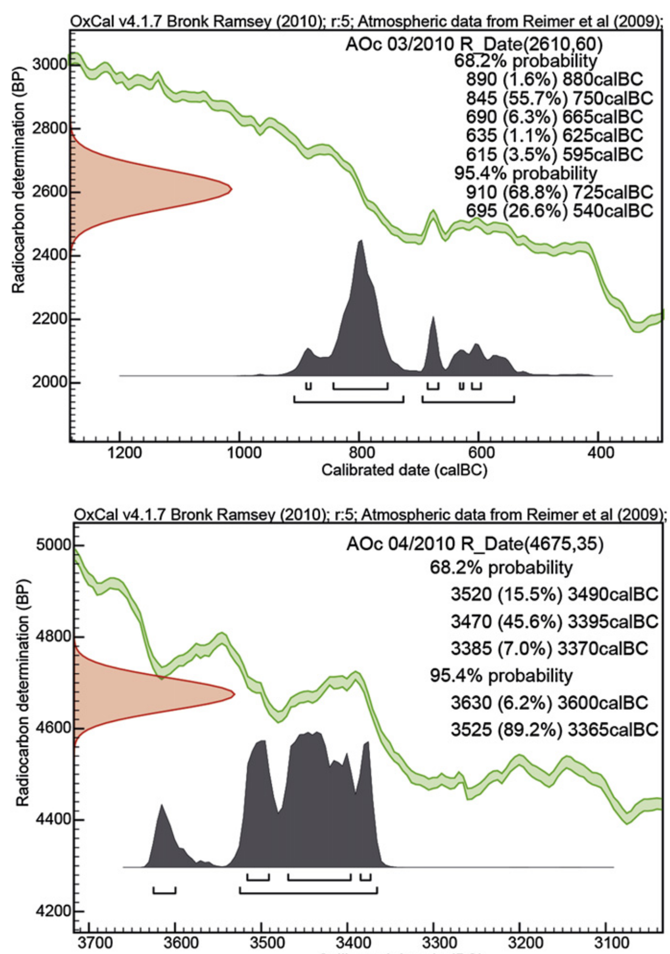

Fig. 10. 1σ and 2σ radiocarbon calibration curves of the two samples dated in Abrigo de los Oculados, produced by OxCal v4.1.7 Bronk Ramsey et al. (2009); *r*:5; Atmospheric data from Reimer et al. (2009).

Table 2
AMS ^{14}C datings of Abrigo de los Oculados (radiocarbon and calibrated ages in bold typeface).

CAMS #	Sample name	$\delta^{13}C$	Fraction modern	±	$D^{14}C$	±	^{14}C age BP	±	Cal BC 2σ
151,646	AOc 03/2010	−9	0.7229	0.0046	−277.1	4.6	**2610**	**60**	**910–540**
151,647	AOc 04/2010	−9	0.5589	0.0023	−441.1	2.3	**4675**	**35**	**3630–3365**

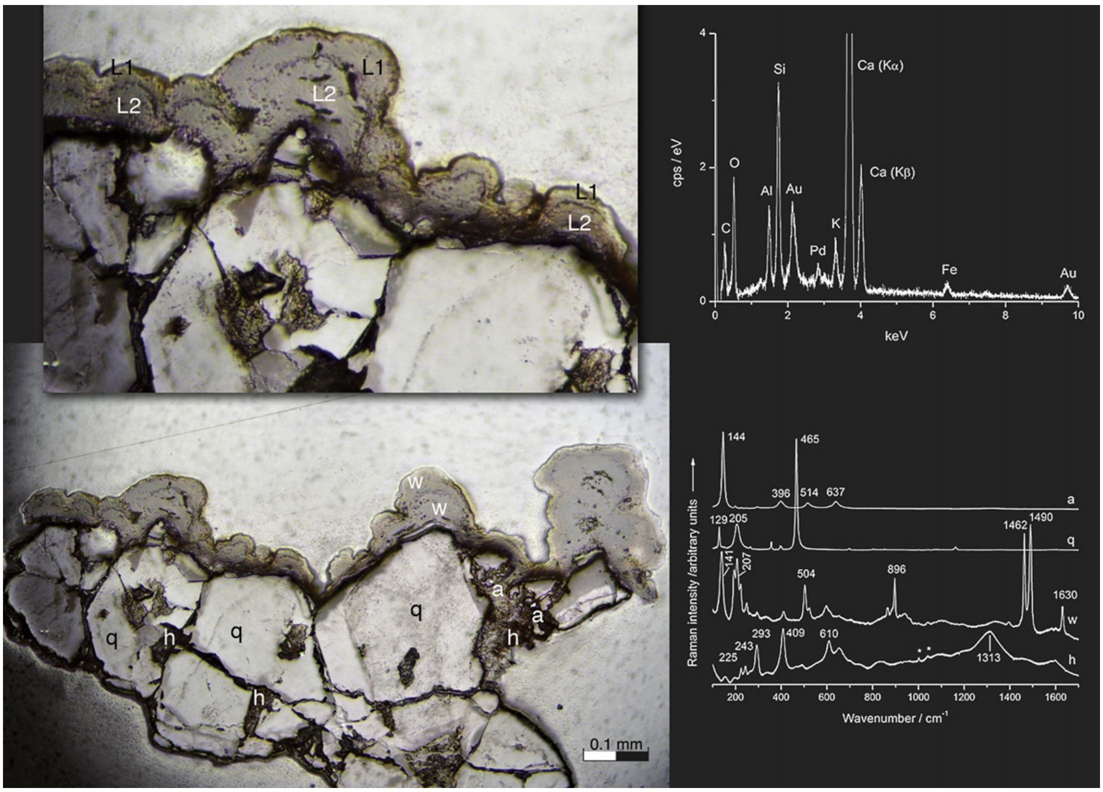

Fig. 11. Microphotograph of a thin section of the substratum and accretion crusts from Abrigo de los Oculados illustrating the microstratigraphy (top). Two accretionary crusts may be distinguished on the rock surface (L1–L2). The composition of the observed coatings was identified by Raman microscopy (right bottom): (a) anatase, (q) α-quartz, (w) whewellite, and (h) haematite (* Bands due to remains of the polyester resin used to prepare the polished thin section). EDX spectrum (right top) of the oxalate layers: C, O and Ca (whewellite) and Si, O, Al, K and Fe (clay minerals, probably responsible of the high level of fluorescence radiation background observed in Raman microscopy), Au and Pd (Au–Pd alloy coating of the thin section to improve the corresponding SEM images).

Table 3
AMS ^{14}C datings of mobiliary eye idols of Spain.

Archaeological site (town, province)	Type of eye-idol	Quant.	BP date	Cal BC date	Bib. reference
Minas de Gavà (Gavà, Barcelona)	Antropomorphic ceramic ware – decoration	1	5190 ± 40 BP–5030 ± 40 BP	4050–3710 2σ	Bosch, 2010
Cueva de los Murciélagos (Zuheros, Córdoba)	Ceramic ware decoration	2		5366 ± 171 cal BC–4775 ± 283 cal BC	Gavilán and Escaceno, 2009
Niuet (L'Alqueria d'Asnar, Alicante)	Long bone eye-idol	3	4490 ± 80 BP	3370–2910 2σ	Pascual and Bernabeu, 1994
Avenc dels Dos Forats (Carcaixent, Valencia)	Long bone eye-idol	1		ca. 2500	Pascual, 2010
Glorieta de San Vicente (Lorca, Murcia)	Painted eye-idol on shoulder blade	1	4075 ± 30 BP	2856–2493 2σ	Martínez et al., 2006
Los Millares I (Santa Fe de Mondujar, Almería)	Several kinds			2930–2570 & 2610–2470	Arribas et al., 1983
Fortín (Santa Fe de Mondujar, Almería)	Several kinds	1		2585–2214	Molina et al., 2004
Almizaraque (Herrerías, Almería)	Several kinds			2500/2400–2100 BC	Maicas, 2007
La Pijotilla (Badajoz, Badajoz)	Antropomorphic eye-idol	1	4130 ± 40 BP	2865–2595 1σ	Hurtado, 2010
La Pijotilla (Badajoz, Badajoz)	Antropomorphic eye-idol	2	4010 ± 80 BP	2836–2368 1σ	Hurtado, 2010
				2865–2296 2σ	Hurtado, 2010
Las Angosturas (Gor, Granada)	Long bone eye-idol	1		2500 ± 140–2030 ± 160 BC	Escoriza, 1990
Las Angosturas (Gor, Granada)	Flat stone eye-idol	1		2500 ± 140–2300 ± 140 BC	Escoriza, 1990
Terrera Venturas (Tabernas, Almería)	Long bone eye-idol	3		2250–1950 cal BC	Gusi and Olària, 1991

Table 4
Oxalate radiocarbon dates in the Sierra de las Cuerdas rock art group.

Reference lab	Site	Sample	$\delta^{13}C$	AMS ^{14}C	±	cal BC 1σ	cal BC 2σ
–	Cueva del Tio Modesto	TMD-1	–	2800	35	1000–910	1050–840
–	Cueva del Tio Modesto	TMD-2	–	6180	35	5210–5060	5230–5010
–	Cueva del Tio Modesto	TMD-3	–	5855	35	4780–4690	4830–4610
133,736	Selva Pascuala, panel 2	SP-84	−25	3490	160	2030–1610	2280–1440
133,737	Marmalo III	M3-85	−25	6955	45	5890–5770	5980–5730
151,646	Abrigo de los Oculados	AOc 03/2010	−9	2610	60	890–595	910–540
151,647	Abrigo de los Oculados	AOc 04/2010	−9	4675	35	3520–3370	3630–3365

The majority of eye-idol figures in the Iberian Peninsula can be inferred to be of Chalcolithic age based on their archaeological contexts. This prehistoric period was also called ‘Bronze I’ until recent times (Almagro, 1973), and is known as ‘Neolithic IIb’ in the Valencian region (Bernabeu, 1989). These different names refer to societies with a fully consolidated production economy, in which lineage memberships were the main mechanisms of social organization. The rise of Chalcolithic societies in the Iberian Peninsula was linked to a new Schematic iconography including the eye-idols, a motif found on a large variety of mobiliary items. They were used in the decoration of pottery, in mobiliary idols and anthropomorphic figurines, and, of course, in rupestrian pictographs and engravings in shelters and deep caves (Fig. 12). Funerary contexts were an important association with this iconography in the Megalithic world, as in the southeastern and western areas of the Peninsula (Leisner and Leisner, 1965; Siret, 1908), and the non Megalithic collective cave burials of the eastern part of Spain (Pascual, 1998).

Eye-idol images are present in the Peninsular archaeological record from the Middle Neolithic to the beginning of the Bell Beaker period (Torregrosa and Galiana, 2001), but they are clearly concentrated from the end of the IVth millennium to the beginning of the IIIrd millennium cal BC (Table 3) (Figs. 13 and 14). Older eye-idols are found in Catalonian and Andalusian locations. At the Catalonian site of Gavà (Barcelona, Spain) an eye-idol was incised on a pottery fragment found in the filling of a well at a variscite

Fig. 12. Iberian mobiliary eye-idols and pictographs. 1- Stelae of San Bernardino; 2- Long bone of Cueva de La Pastora; 3- Long bone of Ereta del Pedregal; 4- Long bone of Niuet; 5- Long bone of Almizaraque; 6- Phalanx idol of Los Castellones (Almería); 7- Antropomorphic idol of Valencina de la Concepción (Sevilla); 8 (a & b)- Symbolic ware of Los Millares; 9- Venus of Gavá; 10- Cilindrical idol (Conquero, Huelva); 11- Rock art - Collado del Guijarral (Segura de la Sierra, Jaén); 12- Rock art - Cueva de la Diosa Madre (Segura de la Sierra, Jaén); 13- Rock art - Cantos de la Visera (Yecla, Murcia); 14- Rock art - Los Órganos (Santa Elena, Jaén). Drawings not to scale.

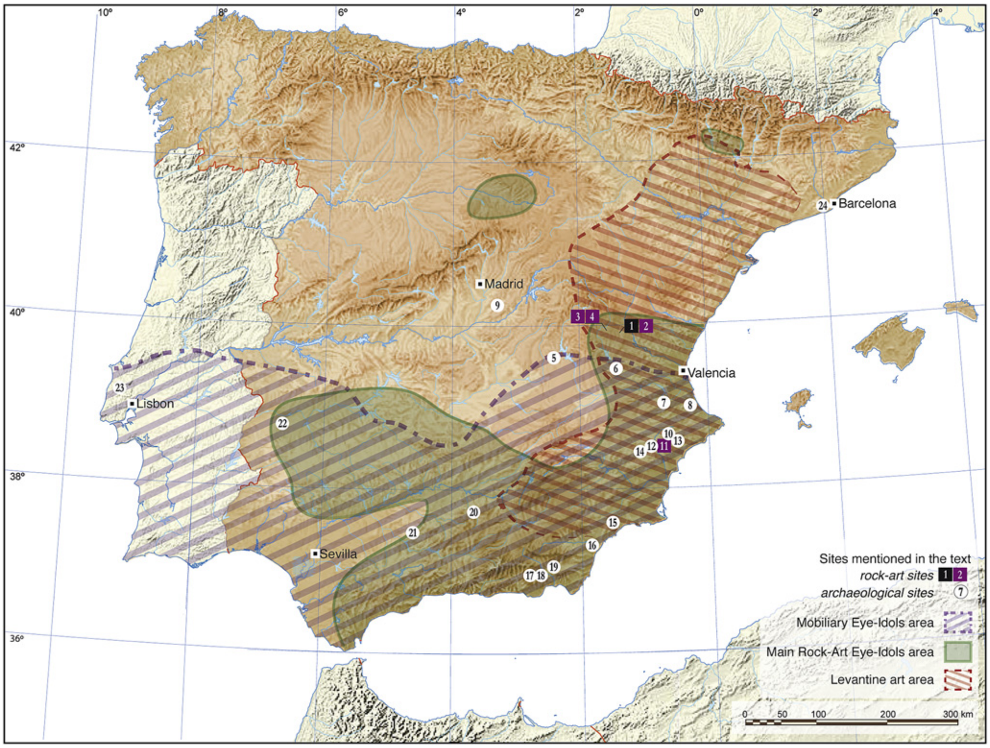

Fig. 13. Map of the Iberian Peninsula with indication of the sites mentioned in the text. 1- Abrigo de los Oculados; 2- Cueva del Tío Modesto; 3- Selva Pascuala; 4- Marmalo III; 5- San Bernardino; 6- Cueva de las Mulatillas; 7- Ereta del Pedregal; 8- Avenc dels Dos Forats; 9- Cueva de Juan Barbero; 10- Cova de l'Or; 11- La Sarga; 12- Cueva de la Pastora; 13- Niuet; 14- El Fontanal; 15- Glorieta de San Vicente; 16- Almizaraque; 17- Los Millares; 18- Fortín I; 19- Terrera Venturas; 20- Marroquíes Bajos; 21- Cueva de los Murciélagos; 22- La Pijotilla; 23- Vila Nova de São Pedro; 24- Gavá.

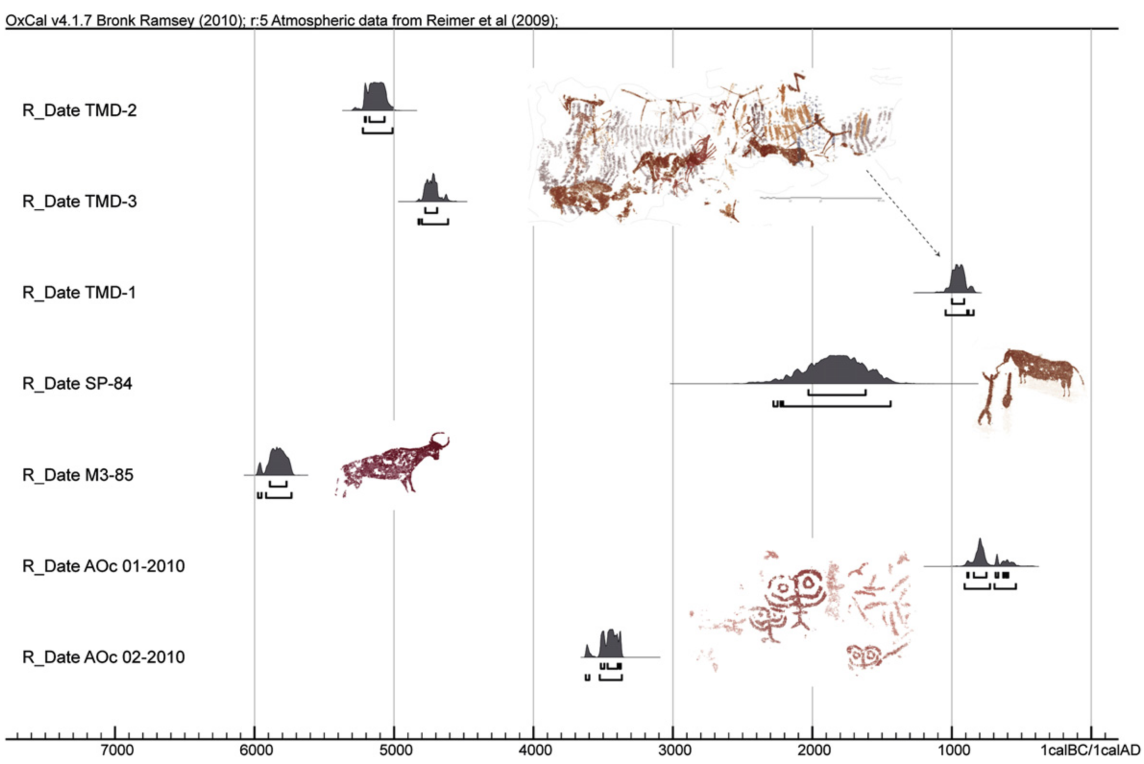

Fig. 14. Graph of 2σ cal BC ages for oxalate crusts dated up to now in Sierra de las Cuerdas (Cuenca) area.

mine (Bosch, 2010). In Andalusia eye-idols are found on incised decorated wares, notably in Cueva de los Murciélagos (Zuheros, Córdoba, Spain), a Neolithic site where eye-idols were also painted on the walls of the cave (Gavilán and Mas, 2006). Similar pottery has been found at other sites in the Guadalquivir valley (Gavilán and Vera, 1993).

Radiocarbon dates related to eye-idols are more numerous in the Late Neolithic and Chalcolithic. In the eastern and southeastern Iberian Peninsula, this iconographic motif is present in the final centuries of the IVth millennium cal BC and up to the beginning of the Bell Beaker horizon, dated 2400–1800 BC. It would still have been in use in the Guadiana valley area and central Spain during the Bell Beaker period (Pascual, 2010). Radiocarbon dates currently published are distributed throughout the IIIrd millennium cal BC, with the exception of a slightly older eye-idol engraved on a long bone recovered at the Niuet site (L'Alquería d'Asnar, Alicante) from silo A of Level II (Pascual and Bernabeu, 1994). These chronologies are consistent with the time frame of Los Millares culture, and with larger sites of the Chalcolithic where many eye-idols have been recovered, such as Vila Nova de São Pedro (Azambuja, Lisbon, Portugal) (Morais Arnaud and Marques Gonçalves, 1995), La Pijotilla (Badajoz, Spain) (Hurtado, 2010) and Marroquíes Bajos (Jaén, Spain) (Sánchez et al., 2005).

Currently, there are no radiocarbon dates for mobiliary eye-idols in the geographic region surrounding Abrigo de los Oculados. The nearest were found at Cueva de las Mulatillas (Villargordo del Cabriel, Valencia, Spain), 50 km south of Sierra de las Cuerdas (Molina and Pedraz, 2000). This site, a Chalcolithic burial cave thought to date to the middle of the IIIrd millennium BC, yielded two eye-idols on long bones. Also in the southeastern Iberian region but further away, at Ereta del Pedregal (Navarrés, Valencia), five eye-idols on long bones were recovered from Neolithic IIb levels (Pascual, 1998). The highest frequency of mobiliary eye-idols has been found in the group of archaeological sites and burial caves of the interior of Alicante, including Cueva de La Pastora, Niuet and El Fontanal (Pascual, 1998). Several kinds of engraved or painted eye-idols have been found at these sites, preserved on long bones.

Eye-idols are less frequent toward the Castilian Plateau, but are present on the stele of Cerro de San Bernardino (La Hinojosa, Cuenca, Spain) (Bueno et al., 1998). This piece differs considerably from the usual typology for these motifs, but was found in the vicinity of Los Dornajos, an archaeological site with chronology extending from the end of the Chalcolithic to the beginning of the Bronze Age (Aceituno et al., 1998). Further away from Abrigo de los Oculados, two eye-idols were recovered on long bones at Cueva de Juan Barbero (Tielmes, Madrid, Spain), a collective burial cave dated to the beginning of Chalcolithic, first half of the IIIth millennium BC (Aliaga, 2008).

Having briefly reviewed the archaeological chronology of mobiliary eye-idols in the Iberian Peninsula, we return our attention to the two radiocarbon dates from Abrigo de los Oculados and their relation with the eye-idols painted there. The *ante quem* date of sample AOc 04/2010, and *post quem* date of sample AOc 03/2010, together bracket the hypothetical age of these figures. The *ante quem* or maximum age indicates they were painted after 3630–3365 cal BC, consistent with the period in which mobiliary eye-idols become more common in the archaeological record. The *post quem* or minimum age, from the overlying oxalate coating, indicates they were painted prior to the 1st millennium cal BC. The latter date is approximately 1000 years past the end of the era of mobiliary eye-idols. We should take into account that the time lapse between the painting event and the beginning of oxalate formation is not known. Moreover, both radiocarbon ages may have undergone some rejuvenation due to possible presence of more recent oxalate in the samples dated.

Agreement has also been found between radiocarbon ages of calcium oxalate coatings and dates based on archaeological contexts in Australia, at the Carpenter's Gap site (Watchman et al., 2005). Oxalate crusts there were shown to be a closed system, not easily alterable. In Brazil, Rowe and Steelman (2003) dated organic material from a red pictograph as well as an oxalate layer that overlay it. Their ages were consistent; the overlying oxalate proved to be younger (2490 $\pm$ 30 BP) than the direct pictograph date (3730 $\pm$ 90 BP). We have also alluded to this possibility in previous papers, because the three radiocarbon ages from Cueva del Tío Modesto (Henarejos, Cuenca, Spain) were internally consistent, reflecting deterioration events of the panel (Ruiz et al., 2006). Additional radiocarbon ages from two sites within the same region, Marmalo III and panel 2 of Selva Pascuala shelter (Villar del Humo, Cuenca, Spain) (Ruiz et al., 2009), further support a similar perspective.

Our results from Sierra de las Cuerdas present a cluster of calcium oxalate radiocarbon ages in agreement with an established archaeological hypothesis (Ruiz, 2006b); that Levantine and zigzag figures are usually older than Schematic ones, and that naturalistic non-Levantine pictographs represent the last stage of painting in this region. In accordance with this hypothesis, the calcium oxalate crusts yielding younger radiocarbon ages are related to Schematic or non-Levantine naturalistic pictographs (samples SP-84 and TMD-1; see Fig. 10 and Table 4), as in Abrigo de los Oculados. Likewise, the older radiocarbon dates were obtained from oxalate layers associated with Levantine and zigzag pictographs (samples TMD-2, TMD-3 and M3-85; see Fig. 10 and Table 4) (Ruiz et al., 2009). All of these radiocarbon dates, except TMD-1, are from oxalate coatings that appear from visual inspection to be covering the nearby pictographs. These samples were removed with careful attention to collect only areas of oxalate crust similar in color to that overlying the pictographs. If our effort to gather uncontaminated specimens availed, these could represent minimum ages for the paintings, even if these crusts suffered any rejuvenation processes. At present, these are the only calcium oxalate radiocarbon dates that can be related to Schematic and Levantine art in the Sierra de las Cuerdas region (Cuenca, Spain).

6. Conclusions

This study carried out at Abrigo de los Oculados could be of some help in the debate on the chronology of post-Palaeolithic open air rock art in Spain. If we are right, it could demonstrate that radiocarbon dates of calcium oxalate accretions may constrain, with an acceptable degree of accuracy, the time period during which pictographs were produced. In this paper, we propose that the age of painted eye-idols at Abrigo de los Oculados may be reasonably inferred from the radiocarbon ages of calcium oxalate layers of this shelter. The archaeological ages of several mobiliary items are in agreement with the oxalate radiocarbon dates bracketing painted eye-idols, although with a wide time range. As long as the stratigraphic relationship between oxalate crusts and a painting event can be established, minimum and maximum ages for pictographs can be obtained.

Multidisciplinary research we have conducted so far has characterized and dated calcium oxalate crusts in sandstone rock shelters in the Sierra de las Cuerdas area. This approach could help to establish an absolute chronological frame of reference for the prehistoric art styles in this area. The same methods could be used regionally with Rock Art of the Mediterranean Basin on the Iberian Peninsula. The utilization of this methodology may help to validate chronological theories currently in use for the three rock painting styles of the eastern part of the Iberian Peninsula. Future research will critically depend on finding sites where oxalate coatings show a clear stratigraphic relationship with pictographs. At the same time, it is necessary to study in greater depth the origin of calcium oxalate coatings, how they form and become associated with rock art.

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Appendix A. Supplementary material

Supplementary data related to this article can be found online at doi:10.1016/j.jas.2012.02.038.

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