

## COVA EIRÓS: AN INTEGRATED APPROACH TO DATING THE EARLIEST KNOWN CAVE ART IN NW IBERIA

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**ABSTRACT.** At Cova Eirós, we discovered 13 panels with paintings and engravings that stylistically point to the final moments of the Upper Paleolithic. Scanning electron microscopy with energy dispersive X-ray spectroscopy and Fourier transform Raman spectroscopy were used to identify charcoal as black pigment. Although contamination from medieval fires inside the cave complicates the dating of these pictographs, analyses of unpainted rock backgrounds allowed calculation corrections for contaminated samples. We used plasma oxidation and accelerator mass spectrometry (AMS) to directly radiocarbon (<sup>14</sup>C) date two charcoal paintings—confirming that the images are more than 9000 yr old. As these paintings superimpose engravings, these <sup>14</sup>C dates also provide a minimum age for an engraving at Cova Eirós that is stylistically Final Magdalenian/Epipaleolithic. This is the first known evidence of Paleolithic cave art in Galicia of NW Iberia.

**KEYWORDS:** northwest Iberia, prehistoric rock art, plasma oxidation, AMS radiocarbon dating.

### INTRODUCTION

We report radiocarbon (<sup>14</sup>C) dates for charcoal cave paintings at Cova Eirós in northwest Iberia. These results support a Paleolithic/Epipaleolithic ascription, providing direct dates for the paintings as well as a minimum age for an engraving located underneath paintings. We used plasma oxidation and accelerator mass spectrometry (AMS) to address important methodological challenges associated with dating rock art (Steelman and Rowe 2012). At Cova Eirós, contamination from medieval fires highlights the importance of taking background control samples of unpainted rock directly adjacent to collected pigment samples. We also conducted environmental scanning electron microscopy with energy dispersive X-ray spectroscopy (ESEM/EDS) and Fourier transform (FT) Raman spectroscopy to confirm the presence of charcoal pigment in the black figures so that small samples (<5 mg) could be collected with minimal impact upon the art.

The combination of plasma oxidation with AMS has been used to date rock paintings worldwide (Russ et al. 1990; Rowe 2001; Steelman et al. 2005; Rowe 2009; Steelman and Rowe 2012; Baker and Armitage 2013; McDonald et al. 2014). Rock art samples present a challenge to traditional <sup>14</sup>C sample preparation methods (acid-base-acid pretreatment followed by combustion), as only a trace amount of organic material is surrounded by an inorganic matrix. The primary advantage of the plasma technique is that the inorganic rock substrate does not decompose during plasma exposure, making extensive acid washes used to remove carbonates prior to combustion unnecessary. Plasma temperatures (<150°C) are below the decomposition temperatures of carbonates and oxalate minerals so that only organic material is extracted for <sup>14</sup>C measurement (Russ et al. 1992). Avoiding acid washes minimizes sample loss during wet

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chemical pretreatment, allowing the analysis of smaller samples. In addition, plasma oxidation is preferable because acid washes may not completely remove oxalates, carbon-containing minerals commonly associated with rock surfaces (Hedges et al. 1998; Armitage et al. 2001). While oxalate mineral accretions can provide minimum and maximum ages for paintings, their inclusion in a dated paint layer would skew results (Steelman et al. 2002).

## **COVA EIRÓS**

Cova Eirós is located in Cancelo (Triacastela, Lugo, Galicia, Spain) on the northwest Iberian Peninsula (Figure 1). Despite being the westernmost area of the Franco-Cantabrian region, northwest Iberia has traditionally remained aloof from Paleolithic art research due to an alleged absence of rock art and scarcity of mobiliary (portable) finds. However, the recurring presence of Upper Paleolithic hunter-gatherer communities in eastern Galicia, evidence of long-range contacts, and the discovery of open-air rock art in Northern Portugal highlighted this anomaly (Fábregas and de Lombera-Hermida 2010; Fábregas et al. 2015). In Galicia, the search for cave art is constrained to narrow bands of limestone formations in the Eastern Ranges with less-developed karst systems compared to other Iberian regions (de Lombera-Hermida 2011). Since no systematic, archaeologically supervised effort to find parietal art has been undertaken in Galicia, we cannot rule out the existence of Paleolithic art even in previously known caves, perhaps containing hardly visible carvings or much-weathered paintings, as happened with recent discoveries in the Cantabrian region (i.e. Montes et al. 2005; Gárate et al. 2014).

At Cova Eirós, archaeological excavation at the cave entrance revealed the existence of several Middle and Upper Paleolithic occupations, as well as Neolithic and Medieval ones (Rodríguez et al. 2011; Rey-Rodríguez et al. 2016). Upper Paleolithic sequences at Cova Eirós range from the Aurignacian (Level 2,  $31,690 \pm 240$  BP, Beta-254280) to the end of the Final Magdalenian (Level B,  $12,060 \pm 50$  BP, Beta-308859). Portable art found in these older levels suggested that Paleolithic parietal cave art might also be found at this site.

During the 2011 field season, we discovered cave art in Cova Eirós. An initial survey of the walls revealed paintings and engravings, stylistically related to the Final Upper Paleolithic/Epipaleolithic (de Lombera and Fábregas 2013; Fábregas et al. 2015). Since then, our research team has conducted a systematic review of the walls and documented 13 panels with 93 motifs: 50% paintings; 46% engravings; and 4% natural rock formations possibly altered to represent zoomorphic silhouettes. Although art has been found everywhere within the cave, images are primarily concentrated on the west wall (Panels I–VI) of the Hall of the Mammoth (Main Hall), the largest chamber at 15 m long by 5 m high. Black charcoal pigment is dominant, with only one instance of red spots on Panel II. On Panel III, the more complex motifs and associations are found, including the concurrence of black paintings and engravings (Figure 2). At other locations in the cave, images are found in alcoves as well as in the southeast gallery (Viñas et al. 2013).

Motifs are usually small, constrained by limited available space on cracked and weathered surfaces, but this seems to reflect a stylistic choice as well. Preservation is poor, either from deterioration (maybe explaining isolated spots or lines) or obstruction by modern graffiti, a blackish film of natural accretions, dust deposition, and smoke from hearths. Engravings are usually thin and shallow, at times forming dense clusters.

As to themes, dots or simple strokes of paint are common and usually placed lower on the panels. These kinds of marks or tracings can be interpreted as either prehistoric graphisms or otherwise unintentional marks, such as evidence of reviving a torchlight, accidental scratches, or topographic

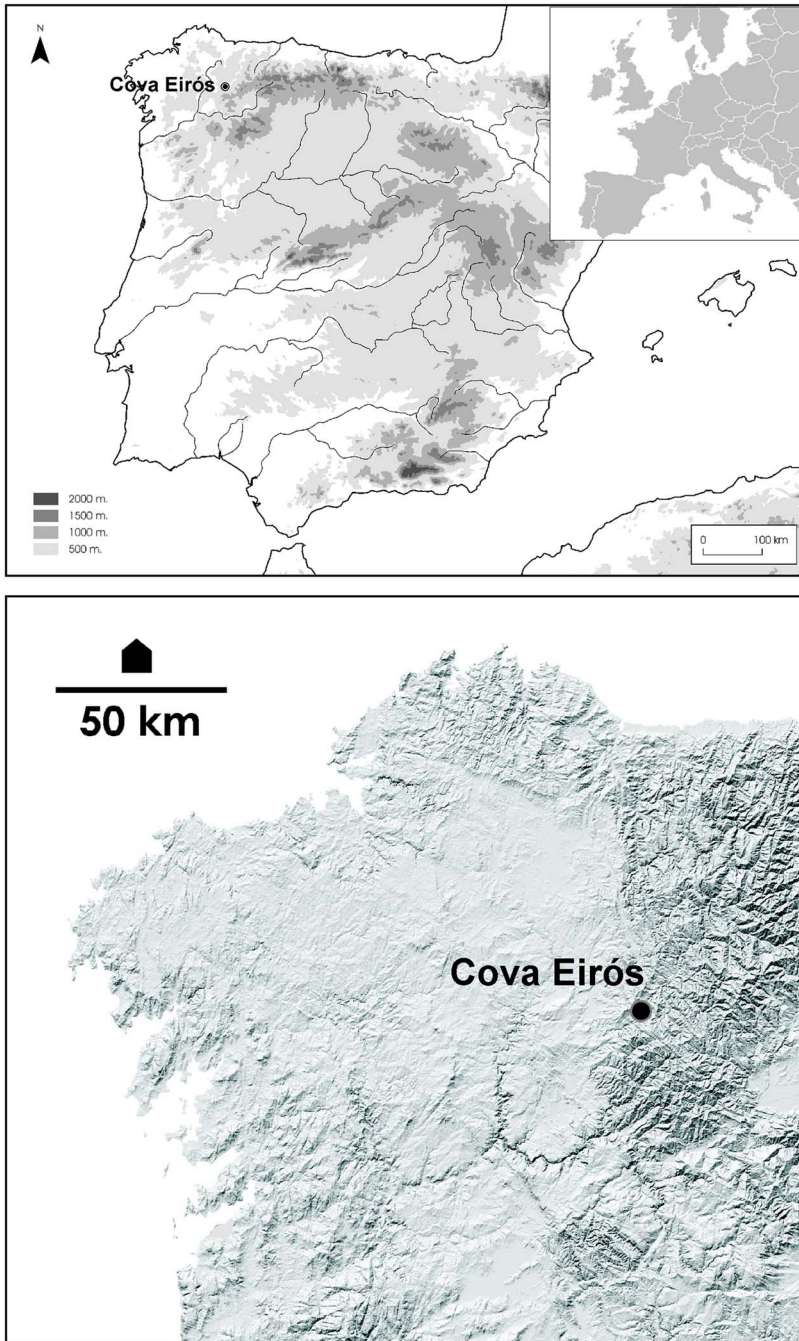


Figure 1 Location of Cova Eirós in NW Iberia.

signals (Diaz 1993), whose creation may have continued until historic times (García and Gonzalez 2003). Also frequent are thin grooves, again isolated or in groups, often appearing as a maze that covers much of the surface. Lastly, there are clearly painted and carved animal representations, generally incomplete (bovids, deer, horses, etc.) (Figure 3).

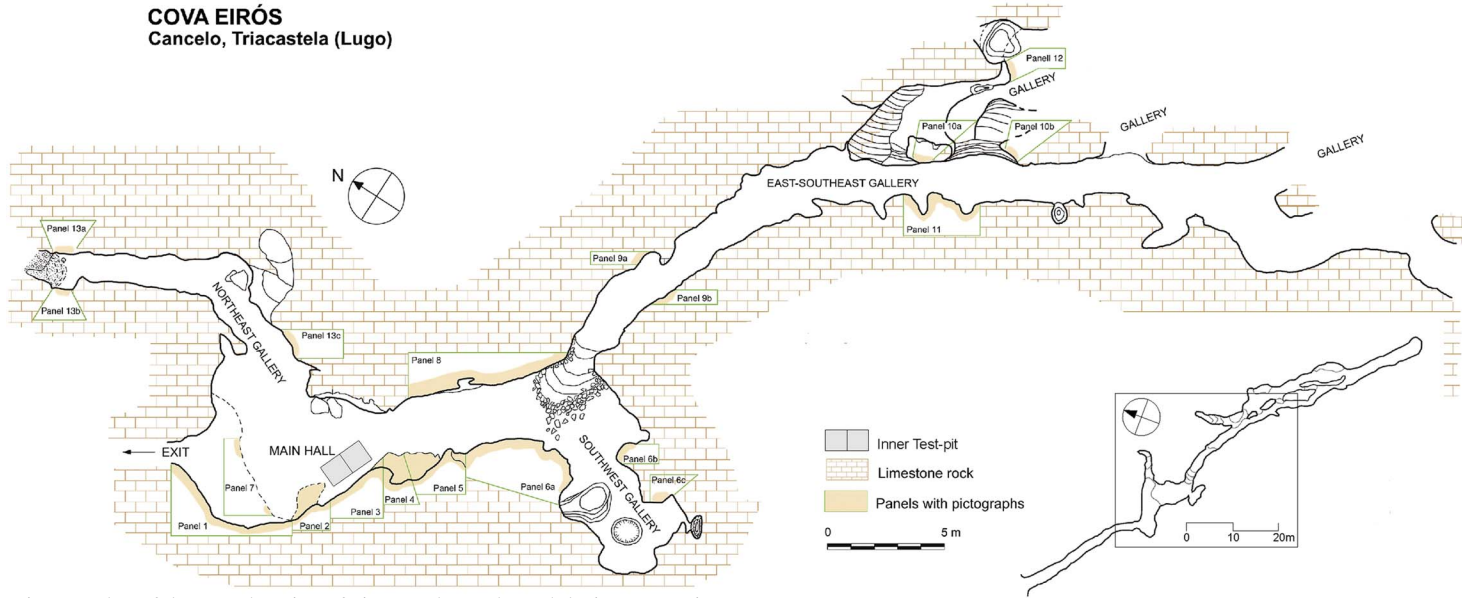


Figure 2 Plan of the cave, location of pictograph panels, and the inner test-pit.

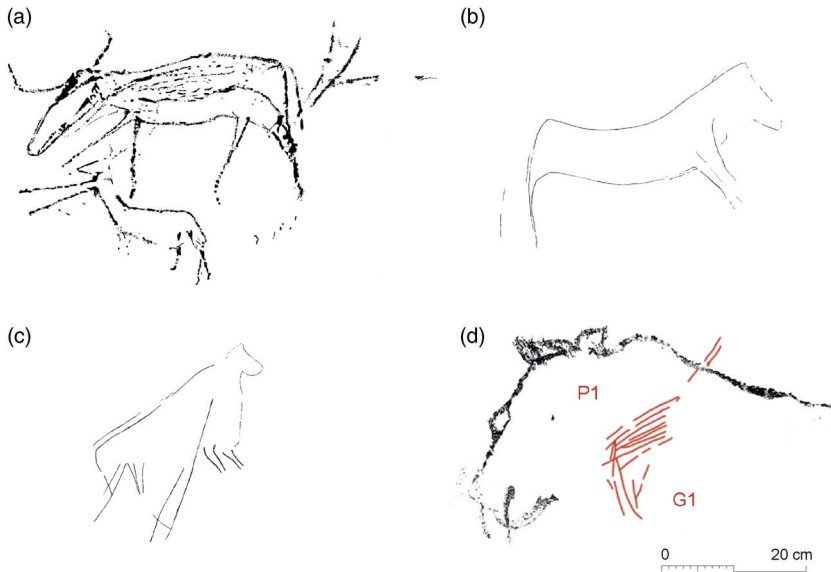


Figure 3 Zoomorphic representations from Cova Eirós. (a) cervid and bovid (Panels I-P5 and P6); (b) equid (Panel XI-G1); (c) bovid (Panel IV-G3); (d) superpositioning of the painted and engraved motifs in Panel III (Panels III-P1 and G1). Sample MD01 comes from the upper trace of the motif Panel III-P1.

Preliminary analyses allow us to put forward a working hypothesis about the chrono-cultural framework of Eirós cave art (de Lombera and Fábregas 2013). Aside from certain multiple and convergent carvings (V- and X-shaped) that might belong to earlier time periods (Gravetian/Solutrean), the more diagnostic examples suggest a final Upper Paleolithic chronology. The two black-painted zoomorphs from Panel I and the engraved motif from Panel III (PIII-G1) with elongated and schematic bodies filled-in with thin lines and simplified representation of the extremities are consistent with the terminal Magdalenian or transition to the Epipaleolithic, as reported elsewhere in Iberia (Alcolea and Balbin 2006; Bueno et al. 2009; Viñas et al. 2010; Collado and García 2010) (Figures 3b and 3d). These traits are also found in the painted figures from the Sala de las Pinturas at Cueva Palomera (Burgos, Spain), AMS dated to  $11,470 \pm 110$  BP and  $10,950 \pm 100$  BP (Corchón et al. 1996), on the engraved plaquettes recovered from the top of level 4 at Rock #1 from Fariseu (Foz Côa, North Portugal), dated by their stratigraphical context between 11,500 and 9800 BP (García and Aubry 2002; Aubry et al. 2009; Mercier et al. 2006; Aubry and Sampaio 2009), and on the zoomorphic representation from Abrigo do Passadeiro (Miranda do Douro, North Portugal), ascribed to the Epipaleolithic (Sanches and Teixeira 2014). These stylistic features are part of the process of geometrization of the figurative component often observed at the Final Magdalenian/Epipaleolithic transition in Iberia (Bueno et al. 2007; García 2013: 505–509).

Radiocarbon samples were collected from Panels I and III (Table 1). On Panel III, a black painting of a zoomorph (PIII-P1, MD01) was drawn on top of an engraved ungulate (PIII-G1) (Figure 3d), suggesting multiple art production events. The stratigraphy and stylistic parallels of the engraving are consistent with a final Magdalenian chronology, reported at the cave entrance (Level B). However, the actual execution of the graphic representations could have a complex history, not necessarily related to the settlement sequence of the site.

Table 1 Description of parietal art and background samples.

	Sample	Motif ID	Description
Painted motifs	MD01	PIII-P1	Upper trace of zoomorph of Panel III
	MD02	PIII-P2	Black spot from lower row of symbol of Panel III
	MD03	PIII-P4	Upper trace of zoomorph of Panel III
	MD04	PI-P1.4	Black spot on a natural relief from Panel I
Backgrounds	5	CE12-GAL-INT-DCH-3	Black deposit covering wall surface of Panel Xb
	6	CE12-MM-P3-N°2	Black deposit covering lower part of Panel III
	7	CE12-P3-N°1	Black deposit covering base of Panel III

Table 2 Radiocarbon dates obtained from excavations.

Location	AMS ID	Sample	$\delta^{13}\text{C}$ (‰)	$^{14}\text{C}$ age (BP)	Reference
Inner test-pit	Beta-356716	Human bone	-19.0	4750 ± 30	
Corridor	Ua-38121	Human bone	-20.8	3151 ± 30	Vidal et al. (2010)
Entrance hearth (UA6)	Beta-308578	Charcoal	-24.1	1040 ± 30	Fábregas et al. (2012)
Inner test-pit hearth	Beta-333971	Charcoal	-22.9	1020 ± 30	
Panel XI (fissure)	Beta-345400	Charcoal	-23.7	1050 ± 30	

Archaeological excavation inside the cave has revealed intense use in later prehistoric and medieval times (Table 2). Human remains were recovered from the inner galleries and are most likely related to burial practices during the Neolithic and Bronze Age (Fábregas et al. 2012). A test pit in the Hall of the Mammoth retrieved the remains of a bonfire dated to 900–1000 AD—just beneath Panel III, coeval with charcoal recovered deeper in the cave from a Panel XI fissure. A hearth and two storage structures at the entrance of the cave also have a similar age (Teira et al. 2012), pointing to intense occupation and exploitation of the cave during this historical period. As there are multiple occupations over long time periods, the aim of this study was to obtain direct dates for the Cova Eirós cave art—within the constraints of extremely small sample sizes that may have been contaminated by medieval fires.

## METHODS

### ESEM/EDS and FT-Raman Spectroscopy

Paint and unpainted rock samples were analyzed with ESEM/EDS and FT-Raman spectroscopy to characterize pigment composition as well as identify possible contaminants and organic materials in the unpainted rock substrate (Serveis de Recursos Científic i Tècnics, URV, Tarragona, Spain) (Viñas et al. 2013). Most samples were from Panel III, which has the largest number of painted and engraved motifs (Table 1, Figure 4). An FEI Quanta 600 ESEM/EDS instrument was used for imaging and elemental analysis, so that no conductive coating was required. Using a Renishaw inVia FT-Raman instrument, the 514 nm line of an argon ion laser was employed for Raman excitation.

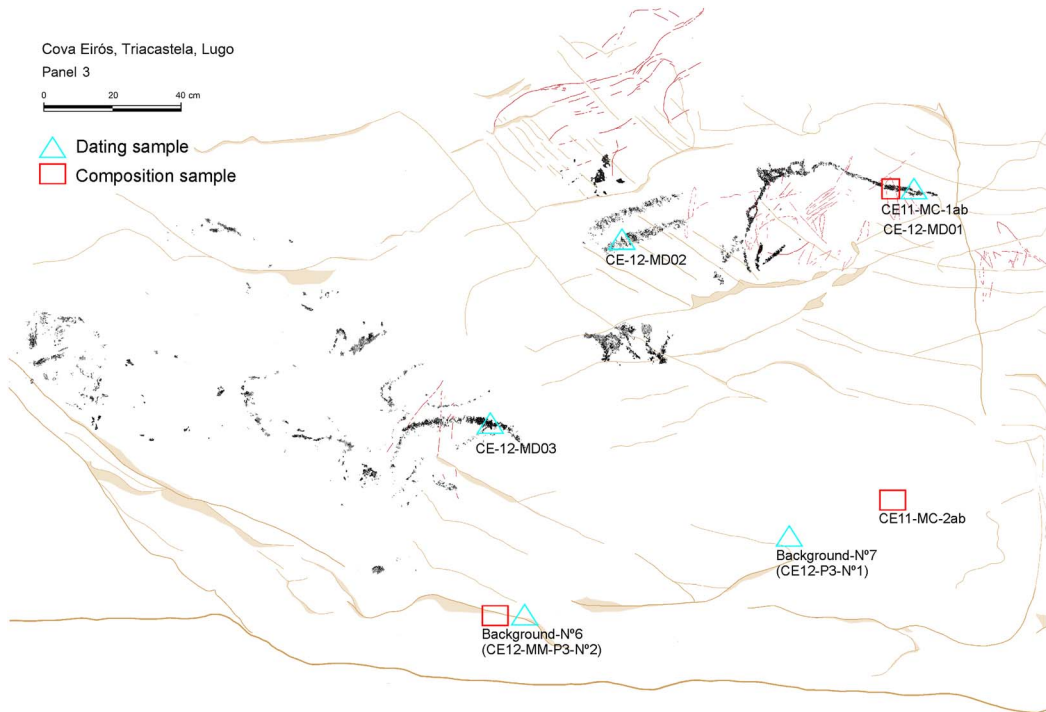


Figure 4 Tracing of painted (black) and engraved (red) motifs from Panel III and location of the samples collected for this  $^{14}\text{C}$  study. (Colors refer to online version.)

Table 3 Uncorrected  $^{14}\text{C}$  results.

Sample	Sample mass (mg)	Carbon ( $\mu\text{g}$ )	$\mu\text{g}/\text{mg}$	UCA ID	CAMS ID	$^{14}\text{C}$ age (BP)
MD01	1.7	60	35	B253	162556	7670 $\pm$ 290
MD02	4.9	20	4	B246	Too small	—
MD03	3.2	50	16	B249	162557	6820 $\pm$ 220
MD04	1.9	20	11	B250	Too small	—
5	123.4	270	2	B264	163373	2465 $\pm$ 40
6	17.7	30	2	—	Too small	—
7	56.6	100	2	B263	163372	1020 $\pm$ 80

### Plasma Oxidation and AMS Radiocarbon Dating

A custom-built radio frequency (RF) plasma oxidation apparatus converted organic material in paint samples to carbon dioxide for AMS  $^{14}\text{C}$  measurement. See McDonald et al. (2014) for a detailed procedure of rock art dating methods employed by the University of Central Arkansas laboratory. We analyzed four black paint samples for dating and three unpainted rock samples as backgrounds to investigate contamination levels of organic material in the rock substrate (Table 1). Paint samples collected for  $^{14}\text{C}$  dating were less than 5 mg, whereas unpainted backgrounds were at least 10 times larger (Table 3). Samples were observed under a stereoscope at  $\times 40$  magnification prior to analysis. Paint samples were black particles with minimal associated rock substrate and no apparent mineral accretion layer. There were also no visible contaminants such as fibers or rootlets observed under magnification. As samples were small, they were ground to a fine powder prior to base pretreatment. All samples were soaked in 1 Molar sodium hydroxide for 1 hr in a  $50 \pm 5^\circ\text{C}$

ultrasonic water bath. After rinsing with deionized water and drying at 110°C, samples were loaded into the glass sample chamber of the plasma instrument. Prior to plasma oxidation, samples were treated with sequential argon plasma exposures at 1 torr argon and 40 W RF for 1 hr to remove adsorbed gases. Then, samples were oxidized with an oxygen plasma exposure at 1 torr oxygen and 100 W RF for 1 hr to convert organic material to carbon dioxide and water. Collected carbon dioxide was sent to the Center for Accelerator Mass Spectrometry (CAMS) at Lawrence Livermore National Laboratory for graphitization and AMS  $^{14}\text{C}$  measurement.

## RESULTS AND DISCUSSION

### ESEM/EDS and FT-Raman Spectroscopy

For the paint samples in this study, no charcoal fragments were observed with ESEM and EDS spectra were inconclusive (Viñas et al. 2013:46–8). In addition, no mineral accretion layer was observed. For charcoal pigment, it is sometimes possible to identify small charcoal fragments in SEM analyses, usually measuring on the order of 10  $\mu\text{m}$  (see Arias et al. 2011; Clottes et al. 1990; Tomasini et al. 2012 for examples). Otherwise, when the pigment is heavily mashed/pounded or when soot is used, those fragments cannot be observed as was the case for the dated samples in this study (Olivares et al., 2009).

However, FT-Raman of the black paint samples confirmed the use of charcoal as pigment. The FT-Raman spectrum for an aliquot of MD01 clearly shows broad bands at 1340  $\text{cm}^{-1}$  (D band) and 1597  $\text{cm}^{-1}$  (G band), related to amorphous carbon of organic origin (Hernanz et al. 2010) (Figure 5). The absence of a band at 961  $\text{cm}^{-1}$ , related to phosphate, discards the possibility of burnt bone pigment (Smith et al. 1999), which is also corroborated by the absence of hydroxyapatite in FTIR spectra (Tomasini et al. 2012). FT-Raman of background samples show a higher concentration of calcite, as well as the presence of organic material and some charcoal particles (Viñas et al. 2013:46–8) (Figure 6).

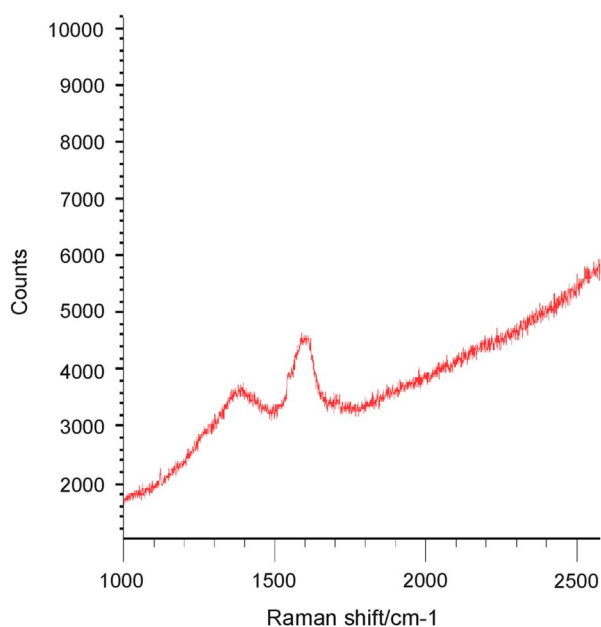


Figure 5 FT-Raman spectrum of sample CE11-MC1ab.

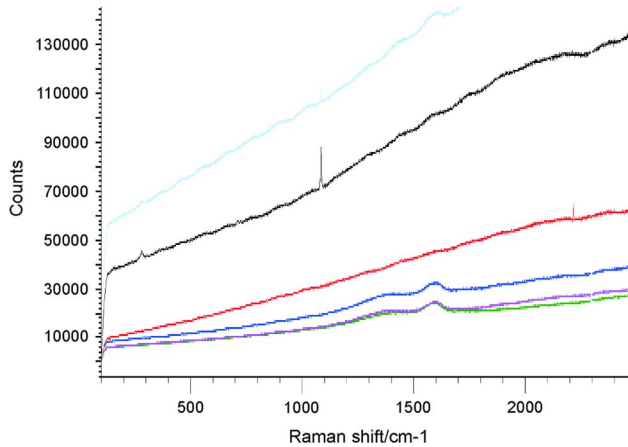


Figure 6 FT-Raman spectra from background sample CE11-MC2ab. Note the bands at  $285\text{ cm}^{-1}$ ,  $711\text{ cm}^{-1}$  and  $1085\text{ cm}^{-1}$ , related to calcite in the upper spectra. The lower ones show the broad bands  $1340\text{ cm}^{-1}$  and  $1597\text{ cm}^{-1}$ , related to carbon in the background sample. The red spectrum shows the high fluorescence of the clays, related to the weathering of the background limestone. (Colors refer to online version.)

## Plasma Oxidation and AMS Radiocarbon Dating

### Paint Samples

Uncorrected age results are shown in Table 3. The  $\delta^{13}\text{C}$  values were assumed to be  $-25\%$ , the value assumed by  $^{14}\text{C}$  laboratories for wood charcoal as carbon samples were too small to split for stable isotope measurements. Only two of the black paint samples from Panel III had sufficient carbon for dating, whereas the others were too small for reliable AMS measurement. During plasma oxidation, the black color of paint samples turned to white ash, further confirming that the paints were made with charcoal. If the pigment had been an inorganic mineral such as manganese dioxide, the black color would have persisted.

### Process Blanks

As age results were younger than expected for these small samples ( $50$  and  $60\ \mu\text{g C}$ ), we  $^{14}\text{C}$  dated USGS coal samples to test accuracy of results. Our laboratory routinely processes standards of known age in the same manner as paint samples to determine the extent of modern contamination incorporated during laboratory procedures. USGS Coal is a “dead”  $^{14}\text{C}$  standard that is millions of years old with virtually no measureable  $^{14}\text{C}$  remaining—an ideal blank for  $^{14}\text{C}$  studies. Frequent evaluation of blanks is necessary in order to determine variability arising from different laboratory techniques and equipment. As sample sizes decrease, the effect of contaminant carbon on blanks is potentially more problematic. From two base-treated USGS coal samples analyzed within a week after the dating of the Cova Eirós paint samples as well as USGS standards regularly analyzed over the past 10 years in our laboratory ( $40$ – $250\ \mu\text{g C}$ ), we introduce  $\leq 1\ \mu\text{g}$  modern carbon during chemical pretreatment, plasma oxidation, graphitization, and AMS measurement. This low background level suggests that minimal modern carbon is introduced during laboratory processing. The average of the two concurrent plasma-oxidized USGS coal samples (CAMS 163261:  $48,270 \pm 270$  BP; CAMS163263:  $48,330 \pm 190$  BP) were used as processes blanks for the AMS laboratory mass balance correction of the ages reported in Table 3.

Table 4 Mass balance contamination and calibration calculations.

Sample	Measured $^{14}\text{C}$ age					Corrected $^{14}\text{C}$ age (BP)	Cal BC (95.4%)	Cal BP (95.4%)
	(BP)	$A_m$	$A_x$	$f$	$A_t$			
MD01	7670	0.3849	0.8792	0.06	0.3533	$8360 \pm 300$		
MD03	6820	0.4278	0.8792	0.13	0.3603	$8200 \pm 310$		
<i>Weighted average</i>						$8280 \pm 220$	7800–6600	9700–8600

### Unpainted Rock Backgrounds

Unfortunately, carbon levels in unpainted rock backgrounds were not negligible suggesting that extracted carbon is a mixture of charcoal from the paint samples and contamination in the rock substrate (Table 3: Samples 5–7). However, paint samples did contain significantly more organic carbon per sample mass. We used the ratio of the  $\mu\text{g}$  amount of organic carbon extracted per mg amount of solid sample as normalized levels of organic carbon present in analyzed samples. For example, all background samples had a  $\mu\text{g}/\text{mg}$  ratio of 2. Using these  $\mu\text{g}/\text{mg}$  values to calculate percent contamination, sample MD01 contained 6% contamination ( $2/35 = 6\%$ ), whereas levels were higher for MD03 at 13% ( $2/16 = 13\%$ ).

This is not surprising as soot from medieval fires was suspected to be present on the cave walls. As stated before, there are two  $^{14}\text{C}$  dates from medieval charcoal inside the cave (Table 2). Charcoal from a small fissure in Panel XI was dated to  $1050 \pm 30$  BP (Beta 345400,  $-23.7\%$ ). Charcoal from a hearth directly in front of Panel III, where the charcoal paintings were sampled, was dated to  $1020 \pm 30$  BP (Beta 333971,  $-22.9\%$ ). Interestingly, carbon in the unpainted rock of background Sample 7 collected from Panel III was  $^{14}\text{C}$  dated to  $1020 \pm 80$  BP (Table 3). This date is statistically indistinguishable from the other two medieval dates for charcoal found in the cave. Using the *R\_Combine* function of the computer program Oxcal, the weighted average of all three medieval dates is  $1034 \pm 21$  BP (980–1030 cal AD) and corresponds to an  $A_x = 0.8792$ .

Background Sample 5 was collected from Panel Xb (in the inner part of the cave) and was dated to  $2465 \pm 40$  BP. This disparate result for another unpainted rock sample shows the variability of organic contamination along the cave expanse and demonstrates the necessity of studying contamination on unpainted rock in as close proximity as possible to dated paintings.

### Contamination Correction

A mass balance calculation was performed to correct  $^{14}\text{C}$  results for the medieval contamination on Panel III, as this is the same location as dated samples MD01 and MD03. The age and contamination equations are  $A = A_o e^{-t/8033}$  and  $A_m = fA_x + (1-f)A_t$ , where  $A$  is the  $^{14}\text{C}$  activity at time  $t$ ,  $A_o$  is the modern  $^{14}\text{C}$  activity,  $A_m$  is the measured  $^{14}\text{C}$  activity,  $A_x$  is the contaminant  $^{14}\text{C}$  activity,  $A_t$  is the true sample  $^{14}\text{C}$  activity, and  $f$  is the fraction of contamination. These calculations are summarized in Table 4.

Using 1034 BP ( $A_x = 0.8792$ ) as the contamination age for Panel III, sample MD01 has a corrected age of  $8360 \pm 300$  BP and sample MD03 has a corrected age of  $8200 \pm 310$  BP (Table 4). These results are statistically indistinguishable, with a weighted average of  $8280 \pm 220$  BP being the best age estimate for the Panel III paintings in the Main Hall. At 95.4% probability ( $2\sigma$ ), the calibrated age range is 7800–6600 cal BC or 9700–8600 cal BP (Table 4). Calibration was performed using the OxCal computer program version 4.2.4 (Bronk Ramsey 2009, 2014) with IntCal13 curve data from Reimer et al. (2013). The associated error for results is large due

to the small size of the samples ( $\leq 60 \mu\text{g}$  carbon from  $< 5 \text{ mg}$ -sized samples), as well as the antiquity of the ages.

While this contamination correction is not completely satisfactory, this is the data we have—admittedly on small samples from a contaminated site. We are in an unfortunate situation where we cannot collect larger samples without damaging the art and there is known contamination from medieval fires at the site. In spite of these challenges, the remarkable agreement between the corrected ages of MD01 and MD03 suggests that this type of background correction may be an appropriate solution for providing an age estimate in this situation.

### *Minimum Age of Cova Eirós Engraving*

Radiocarbon dates for charcoal paintings also provide a minimum age for an engraving that is directly underneath one of the dated paintings. The age result for motif PIII-P1 (MD01) is consistent with its superimposition over the carving PIII-G1, stylistically ascribed to the Final Magdalenian/Epipaleolithic transition, or V Style (Bueno et al. 2007, 2009).

## CONCLUSIONS

To overcome challenges in  $^{14}\text{C}$  dating rock art at Cova Eirós, we utilized an integrated approach: (1) employing SEM/EDS and FT-Raman spectroscopy to identify charcoal as the black pigment; (2) using plasma oxidation combined with AMS  $^{14}\text{C}$  dating; (3) correcting  $^{14}\text{C}$  ages with dated contamination in the unpainted rock substrate to overcome inherent contamination within the paint samples themselves; (4) systematic rock art recording to understand the relationship between paintings and engravings; and (5) stylistic comparisons of the most representative figures at Cova Eirós.

We have recorded 13 decorated panels inside the cave with painted and engraved motifs. Although contamination complicates the dating of these pictographs, the charcoal paintings at Cova Eirós have been shown to be  $\geq 9000$  yr old. Raw  $^{14}\text{C}$  ages were corrected for the percentage of medieval contamination assumed to be present in the paint samples. A weighted average of  $8280 \pm 220$  BP represents the best age estimate for the paintings. Calibration provides a 95.4% probability age range of 7800–6600 cal BC or 9700–8600 cal BP. These results suggest an Epipaleolithic time frame for the two dated paintings at Cova Eirós, consistent with the stylistic ascription of the underlying motif and the most representative/diagnostic images (Style V). Superimpositioning and imagery elsewhere within Cova Eirós suggest that other representations might be much older.

The plasma oxidation method arises as a promising technique to overcome challenges associated with Paleolithic rock art studies. These results are only the second instance that plasma oxidation has been used to date non-Holocene aged materials. Ilger et al. (1995) obtained  $12,180 \pm 125$  BP (AA-9465,  $-25.5\%$ ) and  $11,600 \pm 150$  BP (AA-9766,  $-24.3\%$ ) for charcoal images at La Grotte du Portel in France. A rock art dating intercomparison program based on samples from the Upper Paleolithic site of Chauvet-Pont d'Arc Cave is highly desirable (Quiles et al. 2014). In addition, Pace et al. (2000) and Armitage (1998) demonstrated that base-treated plasma oxidation ages statistically overlap with ABA combustion ages.

Finally, the paintings of Cova Eirós are important because it is the second cave site, along with Cueva Palomera, with Final Magdalenian/Epipaleolithic rock art on the entire Iberian Peninsula. These results are important in establishing a chronological framework for Style V art, which is identified on portable art that has been dated based on indirect stratigraphical methods. The rock art dates for Cova Eirós ( $> 9000$  cal BP) provide an *ante quem terminus* for

the more recent evidence of that style. It is also the westernmost cave with Paleolithic art on the Iberian Peninsula and a crucial link between those well-known cave sites from the Cantabrian region and the recently discovered open-air sanctuaries in NE Portugal. With this find at Cova Eirós, there are opportunities for new discoveries not only in Galician caverns, but also in other rock shelters or open-air outcrops in the region similar to those in nearby territories of the interior of Iberia which have provided akin cultural evidence (valleys of Cõa and Sabor, Siega Verde, etc.) (Baptista and Reis 2009). A systematic survey of sites using modern recording technology (digital photography and enhancement) is necessary in order to record previously unnoticed parietal art. Using an integrated approach to the analysis of Upper Paleolithic hunter-gatherer societies in NW Iberia, our understanding of cultural and socioeconomic relationships and cultural territories in the North Iberia and Cantabrian regions during the Final Upper Paleolithic will be significantly improved.

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## SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit <https://doi.org/10.1017/RDC.2017.4>

## REFERENCES

- Alcolea González JJ, Balbín Berhmann R. 2006. *Arte Paleolítico al Aire Libre: El Yacimiento Rupestre de Siega Verde, Salamanca*. Junta de Castilla y León.
- Arias P, Laval E, Menu M, González Sainz C, Ontañón R. 2011. Les colorants dans l'art pariétal et mobilier paléolithique de La Garma (Cantabrie, Espagne). *L'Anthropologie* 115:425–45.
- Armitage RA. 1998. Radiocarbon dating of charcoal-pigmented rock paintings. [PhD dissertation]. College Station (TX): Department of Chemistry, Texas A&M University.
- Armitage RA, Brady JE, Cobb A, Southon JR, Rowe MW. 2001. Mass spectrometric radiocarbon dates from three rock paintings of known age. *Antiquity* 66:471–80.
- Aubry T, García Díez M, Sampaio J, Plisson H, Chauvière F-X, Tymula S, Calame A, Dechanez I. 2009. Datação das gravuras do Vale do Cõa, In Aubry T, editor. *200 séculos da história do Vale do Cõa: incursões na vida quotidiana dos caçadores-artistas do Paleolítico*. Lisboa: IGESPAR, IP. p 361–477.
- Aubry T, Sampaio J. 2009. Chronologie et contexte archéologique des gravures paléolithiques de plein air de la Vellée du Cõa (Portugal). In: Balbín Berhmann R, editor. *Arte prehistórico al aire libre en el sur de Europa*. Valladolid: Junta de Castilla y León, Consejería de Cultura y Turismo. p 211–23.
- Baker SM, Armitage RA. 2013. Cueva La Conga: First Karst Cave Archaeology in Nicaragua. *Latin American Antiquity* 24:309–29.
- Baptista AM, Reis M. 2009. Prospecção da arte rupestre no Vale do Cõa e Alto Douro português: ponto da situação em julho de 2006. In: Balbín Berhmann R, editor. *Arte prehistórico al aire libre en el sur de Europa*. Valladolid: Junta de Castilla y León, Consejería de Cultura y Turismo, Valladolid. p 145–92.
- Bronk Ramsey C. 2009. Bayesian analysis of radiocarbon dates. *Radiocarbon* 51(1):337–60.
- Bronk Ramsey C. 2014. OxCal version 4.2.4. <http://c14.arch.ox.ac.uk> (accessed April 23, 2014).
- Bueno Ramírez P, de Balbín Behrmann R, Alcolea González JJ. 2007. Style V Dans Le Bassin Du

- Douro Tradition Et Changement Dans Les Graphies Des Chasseurs Du Paléolithique Supérieur Européen. *L'Anthropologie* 111:549–89.
- Bueno Ramirez P, Balbín Berhmann R, Alcolea González JJ. 2009. Estilo V en el ámbito del Duero: Cazadores finiglaciares en Siega Verde (Salamanca). In: Balbín Berhmann R, editor. *Arte Prehistórico al aire libre en el Sur de Europa*. Valladolid: Junta de Castilla y León. Consejería de Cultura y Turismo. p 259–86.
- Clottes J, Menu M, Walter P. 1990. La préparation des peintures magdaléniennes des cavernes ariégeoises. *Bulletin de la Société Préhistorique Française* 87:170–92.
- Collado Giraldo H, García Arranz JJ. 2010. 10.000 años de arte rupestre. El ciclo pre-esquemático de la Península Ibérica y su reflejo en Extremadura (España)”. In: *Global Rock Art. Anais do Congresso Internacional de Arte Rupestre IFRAO, Fundamentos IX*. Sao Raimundo Nonato, Brasil: Fundação Museu do Homen Americano. p 1167–92.
- Corchón MS, Valladas H, Bécarea J, Arnold M, Tisnerat N, Cachier H. 1996. Datación de las pinturas y revisión del arte paleolítico de Cueva Palomera (Ojo Guareña, Burgos, España). *Zephyrus* 49:37–60.
- Díaz Y. 1993. *El arte rupestre esquemático en Cantabria*. Santander: Universidad de Cantabria.
- Fábregas Valcarce R, de Lombera Hermida A. 2010. El Paleolítico superior en Galicia a la luz de las últimas investigaciones. In: Mangado X, editor. *Paleolítico superior peninsular. Novedades del siglo XXI. Homenaje al Profesor Javier Forrea, Monografías SERP, 8*. Barcelona: Universitat de Barcelona. p 255–70.
- Fábregas Valcarce R, de Lombera Hermida A, Serna González MR, Vaquero Rodríguez M, Pérez Rama M, Grandal D'Anglade A, Rodríguez Álvarez XP, Alonso Fernández S, Ameijenda Iglesias A. 2012. Ocupaciones prehistóricas e históricas nas cavidades das Serras Orientais galegas. As covas de Eirós (Triacastela) e Valdivara (Becerreá). *Gallaecia* 31:19–46.
- Fábregas Valcarce R, de Lombera-Hermida A, Viñas Vallverdú R, Rodríguez Álvarez XP, Soares Figueiredo S. 2015. Throwing light on the hidden corners. New data on Palaeolithic art from NW Iberia. In: Bueno-Ramírez P, Bahn P, editors. *Prehistoric Art as Prehistoric Culture. Studies in Honour of Professor Rodrigo de Balbín-Behrmann*. Oxford: Archaeopress. p 171–81.
- Gárate D, Ruiz-Redondo A, Rivero O, Rios-Garaizar J. 2014. El Arte Parietal En El Golfo De Bizkaia: Nuevos Descubrimientos, Nuevas Interpretaciones. In: Medina-Alcaide M<sup>a</sup>, Romero Alonso AJ, Ruiz-Márquez RM, Sanchidrián JL, *Sobre Rocas Y Huesos: Las Sociedades Prehistóricas Y Sus Manifestaciones Rupestres*. p 26–39.
- García Díez M. 2013. La expresión gráfica en La Peña de Estebanvela (Segovia) en el contexto de los últimos grupos de cazadores-recolectores europeos. In: Cacho C, editor. *Ocupaciones magdalenenses en el interior de la Península Ibérica. La Peña de Estebanvela (Ayllón, Segovia)*. Junta de Castilla y León: CSIC. p 472–515.
- García Díez M, Aubry T. 2002. Grafismo mueble en el Valle del Còa (Vila Nova de Foz Côa, Portugal): La estación arqueológica de Fariseu. *Zephyrus* 55:157–82.
- García Díez M, González Morales I. 2003. En torno al llamado «arte Esquemático-abstracto»: A propósito de unas fechas de Covalanas (Ramales de la Victoria, Cantabria). *Veleta* 20:227–41.
- Hernanz A, Ruiz-López JF, Gavira-Vallejo JM, Martín S, Gavrilenko E. 2010. Raman microscopy of prehistoric rock paintings from the Hoz de Vicente, Minglanilla, Cuenca, Spain. *Journal of Raman Spectroscopy* 41:1394–9.
- Hedges REM, Bronk Ramsey C, van Klinken GJ, Pettitt PB, Nielsen-March C, Etchegoyen A, Fernandez Niello JO, Boschin MT, Llamazares AM. 1998. Methodological issues in the <sup>14</sup>C dating of rock paintings. *Radiocarbon* 40(1):35–44.
- Ilger WA, Dauvois M, Hyman M, Menu M, Rowe MW, Vézian J, Walter P. 1995. Datation Radiocarbone de Deux Figures Pariétales de la Grotte du Portel (Commune de Loubens, Ariège). *Bulletin de la Société Préhistorique de L'Ariège* XLIX:231–6.
- de Lombera Hermida A. 2011. Caves and people. Archaeological research at the eastern margins of NW Iberia. In: de Lombera Hermida A, Fábregas Valcarce R, editors. *To the West of Spanish Cantabria: The Palaeolithic Settlement of Galicia, BAR 2283*. Oxford: British Archaeological Reports. p 111–22.
- de Lombera-Hermida A, Fábregas Valcarce R, editors. 2013. *Cova Eirós. Primeras evidencias de arte rupestre Paleolítico en el Noroeste Peninsular*. Santiago de Compostela: Andavira Editora SL.
- McDonald J, Steelman KL, Veth P, Mackey J, Loewen J, Thurber CR, Guilderson TP. 2014. Results from the first intensive dating program for pigment art in the Australian arid zone: insights into recent social complexity. *Journal of Archaeological Science* 46:195–204.
- Mercier N, Valladas H, Aubry T, Zilhão J, Joron J-L, Reyss J-L, Sellami F. 2006. Fariseu: first confirmed open-air Palaeolithic parietal art site in the Còa Valley (Portugal). *Antiquity Project Gallery* 80, 310 (<http://www.antiquity.ac.uk/projgall/mercier/index>) (accessed November 11, 2016).
- Montes Barquín R, Muñoz Fernández E, Morlote Expósito JM. 2005. Hallazgos recientes de arte rupestre paleolítico en la región cantábrica. Los casos de Cantabria. In: Lasheras Coruchaga JA, González Echegaray J, editors. *El significado del arte paleolítico*. Madrid: Ministerio de Cultura. p 77–108.

- Olivares M, Murelaga X, Castro K, Garate D, Hernando C, Corchón S. 2009. Análisis no destructivo de la materia colorante mediante instrumentación Raman portátil en el arte parietal de la cueva de La Peña (Candamo, Asturias). *Excavaciones Arqueológicas en Asturias* 2003-2006:187–92.
- Pace MFN, Hyman M, Rowe MW, Southon JR. 2000. Chemical pretreatment on plasma-chemical extraction for  $^{14}\text{C}$  dating of Pecos River genre rock paintings. *American Indian Rock Art* 24:95–102.
- Quiles A, Valladas H, Geneste J-M, Clottes J, Baffier D, Berthier B, Brock F, Bronk Ramsey C, Delque-Kolich E, Dumoulin J-P, Hajdas I, Hippe K, Hodgins GWL, Hogg A, Jull AJT, Kaltnecker E, de Martino M, Oberlin C, Petchey F, Steier P, Sval H-A, Van der Plicht J, Wild EM, Zazzo A. 2014. Second Radiocarbon Inter-comparison Program for the Chauvet-Pont d'Arc Cave, Ardèche, France. *Radiocarbon* 56(2): 813–50.
- Reimer PJ, Bard E, Bayliss A, Beck JW, Blackwell PG, Bronk Ramsey C, Grootes PM, Guilderson TP, Hafflidason H, Hajdas I, Hatte C, Heaton TJ, Hoffmann DL, Hogg AG, Hughen KA, Kaiser KF, Kromer B, Manning SW, Niu M, Reimer RW, Richards DA, Scott EM, Southon JR, Staff RA, Turney CSM, van der Plicht J. 2013. IntCal13 and Marine13 Radiocarbon Age Calibration Curves 0-50,000 Years cal BP. *Radiocarbon* 55(4):1869–87.
- Rey-Rodríguez I, López-García J-M, Bennàsar M, Bañuls-Cardona S, Blain H-A, Blanco-Lapaz A, Rodríguez-Álvarez X-P, de Lombera-Hermida A, Díaz-Rodríguez M, Ameijenda-Iglesias A, Agustí J, Fábregas-Valcarce R. 2016. Last Neanderthals and first Anatomically Modern Humans in the NW Iberian Peninsula: climatic and environmental conditions inferred from the Cova Eirós small-vertebrate assemblage during MIS 3. *Quaternary Science Reviews* 151:185–97.
- Rodríguez Álvarez XP, De Lombera Hermida A, Fábregas Valcarce R, Lazuén Fernández T. 2011. The Upper Pleistocene site of Cova Eirós (Triacastela, Lugo, Spain). In: de Lombera Hermida A, Fábregas Valcarce R, editors. *To the West of Spanish Cantabria: The Palaeolithic Settlement of Galicia*. Oxford: British Archaeological Reports, Archaeopress. p 123–33.
- Rowe MW. 2001. Dating by AMS radiocarbon analysis. In: Whitley DS, editor. *Handbook of Rock Art Research*. Walnut Creek: AltaMira Press. p 139–66.
- Rowe MW. 2009. Radiocarbon dating of ancient rock paintings. *Analytical Chemistry* 81:1728–35.
- Russ J, Hyman M, Rowe MW. 1992. Direct radiocarbon dating of rock art. *Radiocarbon* 34(3): 867–72.
- Russ J, Hyman M, Shafer HJ, Rowe MW. 1990. Radiocarbon dating of prehistoric rock paintings by selective oxidation of organic carbon. *Nature* 348:710–1.
- Sanches MJ, Teixeira J. 2014. O Abrigo do Passadeiro, Palaçoulo (Miranda do Douro). Um caso de estudo de gravuras rupestres dos inícios do Holocénico no Nordeste de Portugal. *Portvgalia. Nova Serie* 35:61–75.
- Smith DC, Bouchard M, Lorblanchet M. 1999. An initial Raman microscopic investigation of prehistoric rock art in caves of the Quercy district, SW France. *Journal of Raman Spectroscopy* 30:347–54.
- Steelman KL, Rickman R, Rowe MW, Boutton TW, Russ J, Guidon N. 2002. AMS radiocarbon ages of an oxalate accretion and rock paintings at Toca do Serrrote da Bastiana, Brazil. In: Jakes K, editor. *Archaeological Chemistry: Materials, Methods, and Meaning*. Washington DC: ACS Symposium Series. p 22–35.
- Steelman KL, Carrera-Ramírez F, Fábregas-Valcarce R, Guilderson TP, Rowe MW. 2005. Direct radiocarbon dating of megalithic paints from north-west Iberia. *Antiquity* 79:379–89.
- Steelman KL, Rowe MW. 2012. Radiocarbon dating of rock paintings: incorporating pictographs into the archaeological record. In: McDonald J, Veth P, editors. *A Companion to Rock Art (Blackwell Companions to Anthropology series)*. Oxford: Wiley-Blackwell Publishing. p 565–82.
- Teira Brión A, Martín Seijo M, de Lombera Hermida A, Fábregas Valcarce R, Rodríguez Álvarez XP. 2012. Forest resources management during Roman and Medieval cave occupations in the northwest of the Iberian Peninsula: Cova do Xato and Cova Eirós (Galicia, Spain). Wood and charcoal. Evidence for human and natural history. *Sagvntvm extra* 13:159–66.
- Tomasini E, Siracusano G, Maier MS. 2012. Spectroscopic, morphological and chemical characterization of historic pigments based on carbon. *Paths for the identification of an artistic pigment. Microchemical Journal* 102:28–37.
- Viñas R, Rubio A, Ruiz JF. 2010. La técnica paleolítica del trazo fino y estriado entre los orígenes del estilo levantino de la Península Ibérica. In: Clottes J, editor. *Evidencias para una reflexión. Tarascon-sur-Ariège*. Foix: IFRAO Congreso Mundial de Arte Pleistoceno.
- Viñas Vallverdú R, de Lombera Hermida A, Rubio Mora A, Cortón N, López A, Carrera Ramírez F, Rivas Brea T, Rodríguez Álvarez XP, López De Silanes E. 2013. Manifestaciones rupestres. In: de Lombera Hermida A, Fábregas Valcarce R, editores. *Cova Eirós. Primeras evidencias de arte rupestre Paleolítico en el Noroeste Peninsular*. Santiago de Compostela: Andavira Editora SL. p 34–64.