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U-series dating at Nerja cave reveal open system. Questioning the Neanderthal origin of Spanish rock art

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Abstract

U/Th data obtained on CaCO₃ layers covering rock art at Nerja Cave (Spain) evidence erroneous ages and an inverse relation between uranium concentration and apparent ages. This open system behavior could be due to a mechanism causing uranium mobility, resulting in apparent ages being too old with respect to their real age. This article also questions recently published U/Th data on samples from Ardales, Maltravieso and La Pasiega caves (Spain) where a few U/Th ages older than 40 ka BP, suggested that cave art could be attributed to Middle Palaeolithic population (Hoffmann et al., 2018). For these caves, U/Th data also display an inverse relation between U content and ages, indicating possible uranium mobility and erroneous ages.

1. Introduction

Cave art dating is a key issue for the knowledge of human artistic development. Uranium series dating (or uranium-thorium or U/Th) of secondary CaCO₃ layers underlying or overlying rock art has been used when no direct radiocarbon (¹⁴C) dating is applicable in order to give a limit age to the artistic representations (e.g. Bischoff et al. 2003). U/Th data obtained on Spanish decorated caves proposed a significantly older chronology for rock art than the one previously established using archaeological and ¹⁴C studies, and suggested that the Neanderthals may be the authors of the cave art (Pike et al., 2012, Hoffmann et al., 2016, 2018). These data have been intensely discussed from an archaeological and chronometric point of view (e.g. Bednarik et al., 2012, Clottes 2012, Pons-Branchu et al., 2014, Aubert et al. 2018, Slimak et al. 2018, Pearce and Bonneau 2018, White et al., 2019).

In order to test the reliability of U/Th ages obtained on CaCO₃ layers covering rock art, some authors suggested the use of two dating radiometric methods (U/Th and ¹⁴C) on the same samples (e.g. Plagnes et al., 2003, Fontugne et al., 2013). This approach has been done on a few samples from the Lower galleries of Nerja Cave (Spain). In some cases, it revealed age concordance and in other cases age disagreement, interpreted as the consequence of an open

46 system behavior of the sample (unreliable ages, see Valladas et al., 2017) but without
47 determination of mechanism causing it. In this article, we present U/Th data obtained on
48 CaCO₃ samples from the Upper gallery of Nerja Cave, with evidence of open system behavior
49 resulting in erroneous U/Th ages. The recently published old ages obtained for rock art from
50 Spanish caves are also discussed, since the same phenomena could have occurred.

51

52 **2. U/Th dating of CaCO₃ samples from the pisciform chamber at Nerja cave**

53 Nerja cave (Malaga, Spain) is a large cave, developed in Triassic dolomitic marble. It contains
54 important archaeological remains and graphic manifestations including animals and signs
55 (Sanchidrián 1994). ¹⁴C analyses of charcoal fragments indicate human occupation from
56 40,695 to 5,970 yr cal BP (Medina-Alcaide et al., 2015). In the Upper Galleries, the Pisciform
57 Chamber displays a decorated panel with six red paintings, the “Focas”, interpreted as seals
58 (Sanchidrián 1994). From this room, small fragments of CaCO₃ removed from the decorated
59 panel (GN12-10, GN13-20 and GN13-21, GN14-16 see fig. 1a for location of the samples), an
60 active soda straw (GN12-12), two samples (GN13-15 and GN 13-17) taken below and above
61 a charcoal mark, which was already dated by ¹⁴C (Sanchidrian et al., 2017) and a piece of
62 stalactite from the same sector (GN13-18) were studied for U/Th. Sample GN14-16 was
63 separated into two adjacent fractions: GN14-16I (internal part) and GN14-16E (external part).
64 A more detailed samples description is given in SI. Bulk mineralogy of 4 samples has been
65 determined using Fourier Transform Infrared spectroscopy (FTIR).

66

67 U/Th analyses were performed at LSCE (see Sup. Material SI for details on the method and
68 for the table of results), and revealed in the case of the archaeological parietal samples
69 variable uranium (U) contents scattered between 0.15 (GN14-16E) and 9.16 ppm (GN13-20),
70 U isotopic composition close to equilibrium and ²³⁰Th/²³⁸U ratios from 0.014 (GN13-18) up to
71 2.30 (GN14-16E) corresponding to ages ranging from 1.39 ka to out of the U/Th range, and in
72 two cases, no age calculation possible (too high ²³⁰Th/²³⁸U ratios see fig. S3). As expected, the
73 active soda straw (U content of 7.6 ppm) gave a recent age (0.03±0.01 ka). The most striking
74 feature is the inverse relation between ²³⁰Th/²³⁸U ratios and U content (fig. 1b), and the very
75 large U content variability for samples collected in a same area of the cave.

76

77 GN13-15 and GN13-17 (see fig. S1a and b) sampled under and above the charcoal black mark
78 dated from ca 20 to 18 ka cal BP (Sanchidrian et al., 2017) display U/Th ages that are
79 inconsistent with their stratigraphic position (ca. 87 ka BP for the deepest ca 119 ka BP for
80 the surface sample), and with the ¹⁴C age of the charcoal mark indicating biased U/Th ages.

81 For the whole set of data, the inverse relation between U concentrations and ²³⁰Th/²³⁴U ratios
82 indicates a possible U loss for some samples, resulting in an increase of the ²³⁰Th/²³⁴U ratio
83 and consequently of the apparent age. U natural variability (due to water pathways, U source,
84 climate variation, or prior calcite precipitation ...), may in certain cases lead to different U
85 concentrations within secondary CaCO₃ in the same cave. This natural variability could
86 however not explain either the wrong ages obtained for samples GN13-15 and GN13-17
87 (stratigraphic inversion) or the impossibility of conducting an age calculation for samples
88 GN14-16e and GN13-21 (²³⁰Th/²³⁴U >> 1). Mineralogical investigation shows that samples
89 GN13-15, GN13-18 and GN14-16 are mostly composed of calcite crystals while GN12-12 is

90 made of aragonite. The age of this aragonitic sample is the youngest, sub-actual as expected
91 for an active soda straw and has the highest U content of the 4 samples.

92 Such a U loss could be due to leaching by flowing water, mineral alteration, or to
93 mineralogical transformation. In fact, Nerja cave develops in a dolomitic host rock,
94 favourable to the formation of aragonitic speleothems (e.g. Duan et al. 2012). This mineral,
95 with high U contents, has been identified in this cave (de Cisneros et al. 2003, Drugat 2018).
96 Aragonite is a metastable polymorph of CaCO₃ in surface conditions, and tends to transform
97 into calcite, causing U loss and too old apparent U/Th age (e.g. Railsback et al. 2002,
98 Lachniet et al. 2012, Domínguez-Villar et al., 2017, Martín-García et al. 2019). This
99 transformation is likely at Nerja cave and could explain both the inverse relation between U
100 content and ²³⁰Th/²³⁴U ratios and the wrong U/Th ages obtained even if other more complex
101 mechanisms leading to open system behaviour cannot be excluded.

102 **3. Questioning the Neanderthal origin of Iberian rock art**

103 Hoffmann et al, 2016, 2018 and Pike et al. (2012, with U content published in Pike et al.,
104 2017) presented data from La Pasiega, Ardales and Maltravieso Caves (north, south and
105 central Spain). The conclusions of these studies were intensely debated (Bednarik et al., 2012,
106 Clottes 2012, Pons-Branchu et al., 2014, Aubert et al., 2018, Slimak et al., 2018, Pearce and
107 Bonneau, 2018, White and al., 2019). Indeed, as some of the U/Th ages were older than 40
108 kyr, the authors suggested that the rock art in these caves was realized by Neanderthal people.
109 But as shown in figure 2, data from La Pasiega, Ardales and Maltravieso Caves also display
110 an inverse relation between U content and ²³⁰Th/²³⁴U ratios or U/Th ages. The oldest ages
111 always corresponding to low U content.

112 The inverse link observed between U concentration and the age (or the ²³⁰Th/²³⁴U ratios) for
113 the three caves is similar to the one observed at Nerja Cave and can hardly be explained by
114 environmental change. We thus suggest that the oldest published ages for CaCO₃ layers
115 covering rock art from three Spanish caves could be altered by U loss (due to aragonite to
116 calcite transformation, as already observed at Maltravieso Cave by Mancha Flores, 2011, or
117 due to other phenomenon). Further studies in these caves, including mineralogical studies or
118 cross dating could be important for the knowledge of the rock art chronology.

119

120

121 **4. Conclusion**

122 This article highlights the complexity of the dating of parietal carbonate deposits in caves and
123 shows that it is essential to study in more details their formation and evolution in order to get
124 reliable results and establish a robust chronological framework for parietal art.

125 Further analysis should include when possible i) the use of a second chronometer for discrete
126 sampling, ii) mineralogical study in order to identify possible dissolution processes and
127 mineralogical transformations, iii) the analysis of the CaCO₃ layers not only in the part
128 covering rock art, but also laterally in order to understand its variability and check for its
129 preservation, iiiii) analysis of present day CaCO₃ formation.

130

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139

140 **Figure Caption**

141 Figure 1: Location of the CaCO₃ samples on the “Panel de las Focas” from Nerja Cave (left,
142 a). Relation between U content and ²³⁰Th/²³⁸U ratios for the studied samples (right, b). Dots:
143 data from the decorated panel; Squares: samples from the room, near the panel.

144 Figure 2: Relation between U content and ²³⁰Th/²³⁴U ratios (a) or ages (b) for La Pasiega Cave
145 data (Hoffmann et al., 2016: dots; Hoffman et al., 2018: squares). b) relation between U
146 content and ages for Maltravieso (c) and Ardales Caves (c), data from Hoffman et al. (2018).

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