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## Dating of a ring on one of the largest known Roman iron anchors (La Grande-Motte, France): Combined metal and organic material radiocarbon analysis

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2 **Dating of a ring on one of the largest known Roman iron anchors (La Grande-Motte, France):**  
3 **combined metal and organic material radiocarbon analysis**  
4

5  
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## 23 Abstract

24 Underwater operations conducted along the southern French coast have unveiled two large, isolated  
25 anchors of iron. The largest ever found in the ancient Mediterranean, they reveal that Roman  
26 merchantmen moored in Aigues-Mortes Bay. A combination of analyses focusing on the ring, which  
27 belonged to one of the two anchors, offered the opportunity to collect data from isolated anchors and to  
28 document their production. Radiocarbon analysis, conducted for the first time on this type of object,  
29 determined that they were manufactured in the early imperial period. Another key discovery was a layer  
30 of fibers found in a concretion from the ring, which revealed rare remnants of ropes impregnated with  
31 pitch that could correspond to puddening. The replication of similar analyses on rings belonging to other  
32 anchors would provide a better understanding of this crucial component for ancient mooring.  
33

34 keywords: large iron anchors; ring; puddening; radiocarbon analysis; fibers; pitch  
35

## 36 1 Introduction

37 Two iron anchors found in La Grande-Motte (Hérault) reveal significant dimensions. Their discovery  
38 and subsequent examination are not supported by additional archaeological contexts that could assist in  
39 dating. Anchoring is an important topic for historical examination as the manufacturing of these devices  
40 involves advanced skills and many resources, which had significant economic consequences as a result.  
41 So far, earlier studies dedicated to anchors have mostly focused on their material and shape especially  
42 their arms and stock that could produce a chrono-typology (Moll, 1927; Frost, 1970 for the stone-  
43 anchors; Kapitän, 1984; Haldane, 1986a, 1986b; Frost, 1997; Votruba, 2019). More recently, research  
44 has focused on assessing the provenance of ancient anchors, notably through isotopic studies of lead  
45 stock (Kuleff, 1995), and the radiocarbon dating of preserved wood from the same typology of anchors  
46 (Hadas, 2005). Archaeometric studies have been carried out on iron anchors to uncover the  
47 manufacturing methods (among others, Samuels, 1980; Light, 1992; Eliyahu, 2011; Ciarlo, 2011).

48 However, none of these analyses established the dating of the iron anchors by any other parameter aside  
49 from typology. Our paper presents a new methodology to establish dating in order to confirm that the  
50 iron anchors from La Grande Motte are the largest ancient iron anchors to have ever been found. In  
51 addition, the analyses carried out on the ring and its concretion have brought to light probable remains  
52 of fibers, which can be dated to the same period as the manufacturing of the iron anchor.

53

## 54 2 **The anchors: location, finds, context, and characteristics**

55

### 56 2.1 **Location of the anchors**

57

58 The anchors were found in 2016 at La Grande-Motte, a port built in 1965 in an uninhabited place in the  
59 Aigues-Mortes Bay, the easternmost part of the coast before the Rhône delta (Figure 1). The shore is  
60 made up of wetlands and includes numerous lagoons that once connected the ancient settlements  
61 flourishing in the hinterland to the sea with short natural channels called “*grau*.” So far, no evidence of  
62 ancient occupation has been found within the town, unlike the territory bordering the marshes to the  
63 north of the port. The bay located along the coast of Gallia Narbonensis probably belonged to a segment  
64 of an important maritime route. In a favorable location, it was situated between the Roman settlements  
65 of Agatha (Agde), Nemausus (Nîmes), and Arleat (Arles), which constituted significant economic poles.  
66 Even closer to the bay is the ancient harbor of Lattara (Lattes) that reached its peak of occupation in the  
67 1<sup>st</sup> cent. CE (Jorda et al., 2008; Bagan et al., 2010; Steiner et al., 2020). Despite this strategic position,  
68 the bay has not revealed many underwater finds. The most significant ancient underwater remains  
69 discovered in this area were located 5 km away and consisted of drums and lithic building materials  
70 (Jézégou, forthcoming). The stone cargo largely came from a quarry located to the north, in Bois de  
71 Lens (Gard). Actively exploited in the early imperial period (Bessac, 2002), these finds demonstrate that  
72 vessels most likely sailed into this bay from the area north of the marshes.

73

### 74 2.2 **Isolated anchors**

75

76 The magnetometric surveys supervised by M. Guérout (Guérout 2018) that led to the discovery of the  
77 anchors were aimed initially at searching for the wrecks of five Genoese merchantmen that sank in 1165  
78 near the grau de Melgueil. Instead, it resulted in the detection of five iron anchors, including the two  
79 large examples that are the focus of our investigation. They were found isolated and devoid of additional  
80 archaeological contexts such as remains of the hull of a ship or cargo. The three probes (C1, C2 and C3  
81 in Figure 2) uncovered two anchors broken into five fragments lying on the rock seafloor under 0.7 m  
82 of sediment, mainly consisting of sand and silt. Further operations conducted in the bay of Aigues-  
83 Mortes evidenced three more iron anchors to the south deprived of any stock, in addition to a lead stock

84 1.88 m long, belonging to the 3b type as defined by Kapitän (1984) and habitually in use from the 3<sup>rd</sup>  
85 cent. BCE to the 1<sup>st</sup> cent. CE.

86

### 87 2.3 Characteristics of the anchors

88

89 The two large iron anchors are remarkable for their very large size (Table 1). They had been broken  
90 down into five pieces (Figure 3). Pieces C1/1 and C3/1 correspond to the upper part of a shank with  
91 their stocks, also of iron, still set into the latter. We have identified fragments C2 and C3/2 respectively  
92 to be the lower part of the anchors, which included the arms. It is worth noting that the shanks of both  
93 anchors have been fractured at the very same place, 117 and 116 cm from their crowns (all measurements  
94 were taken at the lowest point of the concretions). The fifth piece, which is straight in its general form,  
95 is probably the remnant of a stock (C1/2). Fragments C1/1, C1/2, and C2 came most likely from the  
96 same anchor, whereas C3/1 and C3/2 belong to another example.

97

98 The remnants of the shanks are 375 and 370 cm long (given both shanks are broken). Beyond their very  
99 large size, the two shanks reveal strong resemblances: their rectangular sections are almost square and  
100 of equal dimensions (19 x 17 cm). Their arm-beams are 185 cm in length for the first and 180 cm for  
101 the second. The rings demonstrate corresponding measurements: 32 and 35 cm respectively for the outer  
102 diameter and 24 and 23 cm for the inner diameter. These similarities indicate that the upper and lower  
103 parts of the shank, including its arms, were likely produced using the same technique and could belong  
104 to one or two separate vessels of similar size. Even so, their stocks are quite different: that of the first  
105 anchor is longer than 293 cm with a 11 x 6 cm rectangular section. The length of the second is 228 cm,  
106 and it has a 13 x 12 cm rectangular section. The stocks of both anchors probably slipped inside the  
107 shank, which might explain why they are longer on one side than on the other but a break is not to be  
108 excluded. The lower extremities of the two mooring devices had a crown that does not seem to have  
109 been provided with a second ring (the first and principal was that at the top of the shank). Both anchors  
110 belong to the type B as defined by Kapitän (1984) and were provided with round-shaped arms, which  
111 was a very common typology in the early imperial period.

112

## 113 3 Materials and methods

114 The ring was the key component of the anchor, and thus our present investigation analyzes it carefully.  
115 The C3/1 assembly was pieced together on a barge, and we cut the ring with a circular saw on either  
116 side of the shank (Figure 4). The C3/1 assembly was then re-immersed exactly in its position and re-  
117 sanded according to the DRASSM (Département des Recherches Subaquatiques et Sous-Marines)  
118 instructions. Surprisingly, both cut ends revealed bright surfaces of metal, when the metal within a  
119 concretion is usually found to be far more degraded. After cleaning part of the concretions, a thin black

120 layer mixed with fibers could be observed adhering to the metallic surface. We postulate that this deposit  
121 could correspond to the remains of a rope coated with pitch that was wrapped around the ring, the so-  
122 called puddening, since this is what the shape of the concretion would suggest. Our analysis relied on a  
123 set of techniques conducted on the metal structure of the ring as well as on the black layer and the fibers  
124 that are described below in further detail. The aim was to identify the materials employed and to obtain  
125 the date range using  $^{14}\text{C}$  on the fibers and on the metal by means of an innovative combination of  
126 methods of analysis.

127

### 128 3.1 Radiocarbon dating of the iron from the ring

129

130 Radiocarbon dating of iron produced by the bloomery process is viable since the carbon contained in  
131 the steely zones of the metal came from charcoal used as a fuel during the smelting process (Van der  
132 Merwe and Stuiver, 1968). Within the furnace, the carbon from charcoal and from the CO resulting from  
133 its combustion is incorporated into the metal by means of diffusion. Carbon is hence heterogeneously  
134 distributed within the metallic matrix, ranging from very low contents ( $< 0.02$  wt % C) to higher values  
135 of 0.8 wt % C.

136

137 Several authors have demonstrated that sources of carbon of different origins can contribute to the  
138 misdating of iron (for example, see Craddock et al., 2002, Hüls et al., 2011). By applying an approach  
139 based on accelerator mass spectrometry (AMS), coupled with a metallographic study and an adequate  
140 sampling process, it was recently shown that a reliable radiocarbon dating of the iron can be determined  
141 (Delqué-Količ et al., 2016; Leroy et al., 2015a, 2015b). This study consists of two decisive stages of  
142 investigation in order to document the nature of the metal and to avoid any risks of misdating (recycling  
143 process, presence of exogenous carbon, measurement of a quantity of carbon below the detection limit).  
144 This method aims at determining the nature of the smelting process, identifying the manufacturing  
145 process used by the blacksmith to forge the object (assembly or not of different metal pieces), and  
146 detecting the most carburized areas in the metal to extract enough carbon for the  $^{14}\text{C}$  measurement. Thus,  
147 if this study makes it possible to obtain crucial data on the manufacturing process of the object, a  
148 prerequisite for reliable  $^{14}\text{C}$  dating, it does not seek to characterize the choices and technical gestures  
149 made by the blacksmith or the origin of the metal in detail.

150

151 Full details of the approach in Leroy et al. (2015b), only the most important steps are reported here. A  
152 large specimen was sampled at the less corroded cut end of the ring before being cross-sectioned and  
153 polished with abrasive paper and diamond paste (3 to 1  $\mu\text{m}$  grain size), which are the usual techniques  
154 for the preparation of iron for the exposition of the metallic matrix of the samples. Then, a  
155 metallographic exam of the metal (3% Nital etching, Oberhoffer's reagent) was conducted to reveal the

156 carbon content within the metal, and to visualize the possible welding lines, possibly pointing to the  
157 assembly of different metal pieces and to the possible recycling of old iron.

158

159 The elemental analysis of the Slag Inclusion (SI) entrapped in the metal also assists in providing  
160 pertinent information about the manufacturing of the object, such as the detection of any potential  
161 recycling after the initial smelting process (Leroy et al., 2015b). Indeed, a set of major elemental  
162 compounds of the ore that are not reduced during smelting (mainly  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{K}_2\text{O}$ ,  $\text{CaO}$  and  $\text{MgO}$ )  
163 have in most cases a constant elemental ratio in the SI of each iron artifact (Dillmann and L'Héritier,  
164 2007; Buchwald and Wivel, 1998). The signature of a smelting operation with the same ore, charcoal,  
165 fluxes, and furnace lining can thus be identified through the comparison of the ratios of elements  
166 contained in the SI. When recycled, old iron can be used and welded with other pieces to produce the  
167 final object. The iron artifacts containing separate pieces of different constant ratios can be detected in  
168 their SI and, therefore, various signatures can be identified within a sample. To compare the ratios values  
169 and identify the chemical signature(s) within the sample, we used multivariate analyses (Principal  
170 Component Analysis (PCA) and cluster analysis) following the approach detailed in Disser et al. (2014).  
171 The statistical analysis is based on a scale invariant representation of the element's concentration, which  
172 is obtained by dividing the concentration of all elements by the geometrical mean of the set of measured  
173 elements as the internal standard (Leroy et al., 2012, Disser et al., 2014). The use of multivariate  
174 analysis, such as PCA, is a way to consider simultaneously all the ratios values to detect variabilities  
175 and differences. Prior to sampling for radiocarbon measurements, the compositional investigation of the  
176 SI was thus carried out.

177

178 Following our observations, samples for AMS were collected within the expected highest carburized  
179 zones of the metal with a 3 mm-diameter metallic drill coated with cobalt boron (CoB) to obtain  
180 approximately 1 mg of carbon for  $^{14}\text{C}$  dating. The resulting samples were combusted to  $\text{CO}_2$  according  
181 to the conditions detailed in Leroy et al. (2015b), and the  $\text{CO}_2$  samples were graphitized at the LMC14  
182 laboratory as described in Delqué-Količ et al. (2016). The  $^{14}\text{C}$  measurements were carried out by  
183 "ARTEMIS," an AMS facility located in Saclay (France) (Moreau et al., 2013).

184

## 185 3.2 Analyses and radiocarbon dating of the black layer and fibers

186 Very small fibers were visible within the black layer, and examination under a digital microscope (Dino-  
187 Lite AM7915MZTL) confirmed the presence of submillimetric fibers impregnated with a brown to dark  
188 matter (Figure 5).

189

### 190 3.2.1 Fiber preparation for SEM analysis

191

192 The microbotanical remains were investigated with the scanning electron microscope (SEM) Neoscope  
193 Philips XL 30 CP. As the remains suffered a mineralization process, the samples were placed on an  
194 adhesive carbon disc without any further action. The SEM provides high resolution pictures of up to a  
195 nanometer of the surface of the sample surface and is therefore an effective tool to obtain the basic data  
196 for establishing a taxonomic identification.

197

### 198 **3.2.2 Analytical procedure for the organic molecular investigation of pitch and fibers**

199

#### 200 **3.2.2.1 Extraction with organic solvents**

201 A small quantity of the black layer (984 mg) visible within the concretion was collected by scraping  
202 with a metal spatula. The sample was extracted by sonication (20 min) with a mixture of  
203 dichloromethane (DCM) and methanol (MeOH) (1:1 v/v; 30 ml). The solvent extract (22.5 mg) was  
204 recovered after centrifugation and removal of the solvents under reduced pressure.

205

#### 206 **3.2.2.2 Derivatization and fractionation of the organic extracts**

207 The organic extract was acetylated with a mixture of pyridine/acetic anhydride (1:1 v/v, 400  $\mu$ l; 1 h, 60  
208 °C). Following the addition of MeOH (1 mL), the solvents and the excess of reagent were removed  
209 under a flow of argon. The acetylated extract was treated with N,N-dimethylformamide dimethylacetal  
210 (150  $\mu$ L) in toluene (1.5 ml) at 70 °C for 3 h in order to methylate the carboxylic acids. The formation  
211 of a dark precipitate was observed. The solvent-soluble part was recovered with a Pasteur pipette and  
212 the solvents were removed under a flow of argon, yielding the derivatized organic extract (16.5 mg).  
213 After treatment with activated copper to remove elemental sulfur, the derivatized extract was  
214 fractionated on a silica gel column eluting with 3 dead volumes of a mixture of DCM and ethylacetate  
215 (EtOAc) (8:2, v/v) to yield an apolar fraction (1.1 mg) analyzed using gas chromatography coupled to  
216 mass spectrometry (GC-MS).

217

#### 218 **3.2.2.3 GC-MS**

219 GC-MS analyses were performed on a Thermo Scientific Trace Ultra gas chromatograph equipped with  
220 a programmed temperature vaporizing injector coupled to a Thermo Scientific TSQ Quantum mass  
221 spectrometer. The source was set at 220 °C and the mass spectrometer operated in the electron ionization  
222 mode at 70 eV and scanning  $m/z$  50 to 700. Compound separation was performed on a HP5-MS column  
223 (30 m x 0.25 mm, 0.1  $\mu$ m film thickness) using He as the carrier gas (constant flow, 1.1 mL/min). The  
224 oven temperature program was 70 °C (5 min), 70 °C – 240 °C (4 °C/min), 240 °C – 300 °C (10 °C/min),  
225 and isothermal at 300 °C (20 min).

226

### 227 **3.2.3 Preparation for radiocarbon dating of plant fibers**

228 Fibers for dating were separated with tweezers from the sample collected within the concretion covering  
229 the anchor ring after extraction with organic solvents, (cf. 3.2.2.1). The fibers were then subjected to an  
230 acid-base-acid chemical cleaning in the Laboratoire de Mesure du Carbone 14. This treatment eliminates  
231 potential contaminations from carbonated and humic origin. After drying, 12.3 mg of clean sample were  
232 combusted at 835 °C for 5 h in the presence of 500 mg of CuO grains and an Ag wire. The pure CO<sub>2</sub>  
233 evolved from the combustion step was then reduced into graphitic carbon that was pressed to form a  
234 target for the <sup>14</sup>C measurements in the AMS facility (Dumoulin et al., 2017 and Moreau et al. 2013).  
235 The radiocarbon result was calibrated with the OxCal 4.4 software (Bronk Ramsey, 2009) using the  
236 IntCal20 calibration curve (Reimer et al., 2020). An amount of 0.66 mg of carbon could be extracted by  
237 combusting the sample, representing about 5% carbon content. Its relatively poor carbon content is  
238 related to the highly mineralized context characterized by concretion and metal.

239

## 240 **4 Results**

241

### 242 **4.1 Analyses of the ring**

#### 243 **4.1.1 Nature of the metal and manufacturing process**

244 The metallographic study of the alloy matrix evidenced a homogeneous microstructure with very low  
245 carbon content (< 0.02 wt% C) on the whole surface of the cross-section (Figure 6). The composition of  
246 the slag inclusions obtained from the chemical analysis is shown in the supplemental material (Table  
247 S1). The multivariate treatment of the chemical data, which are the ratios of elements notified X<sub>ij</sub> in  
248 Figure 7, revealed two distinct chemical signatures indicative of the use of metal pieces from different  
249 smelting operations, and potentially from two different workshops, that were welded together. The  
250 distinction between signatures is more specifically linked to differences in ratios including CaO and  
251 K<sub>2</sub>O. The presence of two chemical signatures also supports the presence of a welding line that is hardly  
252 visible at the microscopic scale (Figure 7), and thus may reflect a particularly skilled smithing  
253 production.

254

255 The SI also contains significant phosphorus (P<sub>2</sub>O<sub>5</sub> weighted average content of 1.6% and 1.2% for each  
256 iron piece), indicating the presence of this element in the metal that was not revealed by etching with  
257 the Oberhoffer's reagent (absence of "ghost structures"). This phosphorus content, even when low, could  
258 have had direct consequences for the behavior of the metal by making ferritic alloy less ductile (Stewart  
259 et al., 2000). It could also explain the low C content in the metal, as phosphorus hinders the incorporation  
260 of C into the iron (Buchwald, V. F., 2005). While the presence of phosphorus is usually not favorable  
261 for iron, it cannot be excluded that the use of this metal quality was suitable for manufacturing the



262 massive ring of the anchors, which needed to be sufficiently robust, as it was the principal component  
263 connecting the anchor to the ship.

264

265 The phosphorus content in the SI may also evidence the use of a rather phosphoric ore source for the  
266 smelting process. Phosphoric ore deposits are present in many active siderurgical places in the western  
267 Roman Empire (Pagès et al., 2022; Kaloyeros and Ehrenreich, 1990), and a future study on provenance  
268 where we would compare chemical signatures of traces elements would allow for the testing of various  
269 hypotheses regarding its origin.

270

#### 271 **4.1.2 Radiocarbon dating of the ring**

272 The ferritic nature (< 0.02 wt % C) of the alloy does not usually allow a radiocarbon study of iron since  
273 no carburized zone was found. Nevertheless, the large size of the sample allowed us to take the required  
274 quantity (4–5 g for an alloy of 0.02 wt % C) in order to extract the expected minimal amount of carbon  
275 required. At this stage of investigation, it is not excluded either that recycled scrap iron (i.e., older iron)  
276 was used and welded with other pieces of metal to manufacture the object since at least two different  
277 metal pieces were detected in the sample. If it is not necessarily so, one  $^{14}\text{C}$  sample was taken from  
278 within each metal piece (4 g each) to avoid mixing samples of possibly different dating. Quantities of  
279 carbon (0.3 mg and 0.1 mg respectively) were finally measured (Table 2). The very small mass of carbon  
280 led to a greater uncertainty for sample 62203. Overall, the two radiocarbon dates are fully coherent and  
281 can be dated to the 2<sup>nd</sup> century CE. This result also shows that the two metal pieces from different  
282 workshops are contemporary and that no recycling case can be detected from this investigation.

283

## 284 **4.2 Analyses of the black layer and fibers**

### 285 **4.2.1 Fiber identification**

286

287 Phytoliths (silica bodies produced by plants) were observed for most of the SEM pictures together with  
288 other anatomical features, such as epidermal cells with sinuous walls and simple pits (Figure 8a). These  
289 indicate that the remains came from a leaf or a stem of a monocotyledon plant (i.e., from a plant that  
290 does not produce wood). The long cells with sinuous walls are common to Gramineae, such as *Stipa* sp.,  
291 Juncaceae and Cyperaceae (Gale and Cutler, 2000). The triangular shape of the remains seen in a  
292 transverse section is a diagnostic feature of Cyperaceae (Metcalf, 1969). The stem and leaf epiderms  
293 of Cyperaceae such as *Cyperus* spp. or *Carex* spp. exhibit some short and long cells morphology closely  
294 related to the phytolith shape that could be seen preserved on the ring (Hameed et al., 2012). Other  
295 epidermal characters were observed, namely paracytic stomata (two guard cells), trichomes (hairs), and  
296 phytoliths of conical shape (Figure 8 b). Above all, the ridge-shaped phytoliths described by Metcalfe

297 (1971) were observed. Stevanato et al. (2019) more recently detected and described this phytolith shape  
298 on 11 genera of Cyperaceae under the naming cylindrical sulcate tracheids.

299

300 A phytolith reference collection, including Cyperaceae, was documented by Fernández Honaine et al.  
301 (2009). Many Cyperaceae phytoliths are consistent with the microbotanical remains that we observed  
302 on the ring. Their anatomical features are similar to those that appear in the University College of  
303 London's online reference collection (Phytolith taxa index), which include *Scirpus lacustris*. The fibers  
304 preserved on the ring most likely originate from a Cyperaceae stem or leaf. This family is well known  
305 for its fiber properties whose genera include *Carex* and *Cyperus*. Unfortunately, it is difficult to  
306 determine further the nature of these remains as most of the characters described above are shared at the  
307 family level.

308

#### 309 **4.2.2 Organic molecular investigation of the pitch and fibers**

310

311 This molecular investigation aimed at determining whether the fabrics or ropes used for the puddening  
312 of the ring were impregnated with an organic substance, such as a resin or a pitch-based material,  
313 commonly in use ("tarring") in marine environments (e.g., Bailly, 2015). In addition, this examination  
314 provides further information concerning the botanical origin of the fabrics or rope used for the  
315 puddening of the ring. Thus, the lipids from the apolar part of the derivatized solvent extract of the  
316 sample were investigated using GC-MS.

317

318 The presence of diterpenoid biomarkers related to abietane (H1-5, A1, Figure 9) suggests that the  
319 organic substance corresponds to a resin or a pitch originating from conifers, specifically Pinaceae (e.g.  
320 Evershed et al., 1985; Colombini et al., 2003; Connan et Nissenbaum, 2003; Bailly, 2015, Bailly et al.,  
321 2016). These substances were commonly used to impregnate fabrics and ropes for the puddening of the  
322 anchor ring. The importance of mono- to triaromatic diterpenoid hydrocarbons, such as H1-4 relative to  
323 resin acids like A1 and the occurrence of methyl retene H5, suggests that this organic substance had  
324 undergone significant thermal stress. It can therefore be proposed that it corresponds to a conifer tar  
325 (i.e., pitch) and not to a resin (e.g., Evershed et al., 1985; Colombini et al., 2003; Connan et Nissenbaum,  
326 2003; Bailly et al., 2016).

327

328 However, the sole presence of these aromatic compounds is generally not sufficient to demonstrate that  
329 the organic substance analyzed is a tar rather than a resin. Indeed, the majority of these compounds can  
330 also be formed by the diagenetic transformation of diterpenic acids (e.g., Simoneit et al., 1986; Reunanen  
331 et al., 1990; Martin et al., 1999). Nevertheless, a ratio of H1 relative to H2 of ca. 3/4 as observed in this  
332 case, corresponds to that generally found in pitch and not when H1 and H2 are formed by anaerobic  
333 diagenetic processes (large predominance of H2; Hynning et al., 1993; Tavendale et al., 1997; Martin et

334 al., 1999; Bailly, 2015; Bailly et al., 2016). In addition, the methylated analogues of retene, such as H5,  
335 are frequently encountered in pitch and are not formed by early diagenetic transformation processes  
336 affecting resin acids (Bailly et al., 2016). The extremely low amounts of diterpenoids observed within  
337 the concretion that developed around the anchor ring could be explained by the destruction of most of  
338 the original organic material due to the unfavorable diagenetic conditions for preservation prevailing in  
339 the concretion.

340  
341 The main compounds eluted at the end of the chromatogram can be divided into four main series  
342 comprising linear compounds (empty and filled triangles, Figure 9), steroids (S1, S2), triterpenes (T1-  
343 6), and phenolic derivatives (filled diamonds). These compounds are all typical biomarkers originating  
344 from land plants. *n*-Alkanes and *n*-alcohols are constituents of cuticular waxes widely distributed in the  
345 plant kingdom and are therefore not specific biomarkers. Nevertheless, their distribution can sometimes  
346 be interpreted in terms of botanical origin based on homologue predominance (e.g., Van Bergen et al.,  
347 1997; Trendel et al., 2010). Steroids are dominated by C<sub>29</sub> sterols and stanols (S1, S2), which are typical  
348 plant biomarkers. The main triterpenes identified (T1-T6) are common and widely distributed in  
349 angiosperms and cannot be related to a more specific source. Finally, the phenolic compounds could not  
350 be unambiguously identified solely based on mass spectrometry and might be related to lignin  
351 derivatives.

352  
353 All these compounds most likely originated from the fibers derived from the ropes used for the  
354 puddening of the ring and correspond to lipids from angiosperms. The distribution of these compounds  
355 is thus not incompatible with that expected for plants of the *Cyperaceae* family (see section 3.2.3). Yet  
356 they do not possess a specificity sufficient to relate them to a more precise botanical origin at the species  
357 or genus level. In addition, it is difficult to find in the literature comparative molecular distributions of  
358 organic extracts of sedges that might have been used in such a context. It appears that hemp can  
359 unambiguously be excluded as a possible vegetal source. Indeed, if the predominance of the C<sub>28</sub>  
360 homologue among the *n*-alcohols is a typical feature of the organic extract of hemp ropes as is the  
361 presence of triterpenoids T1-T4 (Gutierrez et al., 2006; Bailly, 2015), the predominance of the C<sub>31</sub>  
362 homologue among the *n*-alkanes from our sample is not compatible with this possibility since the C<sub>29</sub>  
363 homologue predominates the *n*-alkane distribution from hemp extracts (Gutierrez et al., 2006; Bailly,  
364 2015).

365

#### 366 **4.2.3 Radiocarbon dating of plant fibers**

367 After calibration, a radiocarbon date of 1840 +/- 30 BP obtained from the sample gave two calendar  
368 intervals of 124–250 cal CE with 91.8% confidence and 295–310 cal CE with 3.6% confidence (Table  
369 4). The range 124–250 cal CE is closer to the iron radiocarbon ages, which first confirms the  
370 chronological consistency of the set ring/puddening. Assuming a possible old-wood effect for the

371 radiocarbon dating of the iron, this range can be suggested for both the manufacture of the iron and the  
372 organics used for puddening, Concerning the anchor itself, the chronological data obtained for the ring  
373 and the puddening allows us for the determination of the date of its production before the 3<sup>rd</sup> century  
374 CE.

375

## 376 5 Discussion

377

378 The focus on the ring has brought forth significant material and evidence contributing to larger  
379 discussions about the technology of production of these large devices. The radiocarbon dating of both  
380 the carbon content of the wrought iron and the fibers (Figure 10) indicates that it was produced in the  
381 early imperial period. The proximity and similar morphology of the anchors invites us to consider that  
382 the dating of the ring was the same for both anchors. This dating fits well with their typology (Kapitän,  
383 1984) and removes doubts regarding the ancient origin of these out-of-context finds. They were forged  
384 at a time when iron anchors progressively replaced wooden anchors with fixed lead stock (Gianfrotta,  
385 1980; Sadania, 2017). Iron anchors with a removable stock probably saved space on ships, which was a  
386 clear advantage (Kapitän, 1984; Haldane, 1986a).

387

388 The two Roman iron anchors in La Grande Motte seem to be the largest to have ever been found. Out  
389 of the 354 iron anchors reported by Votruba (2014), only five of them measure over 300 cm. The most  
390 famous of this group was certainly the anchor found in Lake Nemi (35/47 CE). Covered by wooden  
391 sheathing, its shank is 361 cm long, and the object weighs 417 kg (Ucelli, 1950). A 350 cm-long iron  
392 anchor was also found on the starboard aft of the Sud-Lavezzi 2 shipwreck (Liou and Domergue, 1990).  
393 Its 220 cm-long iron stock still set into the shank indicates that the anchor was in use when the ship sank  
394 in the early 1<sup>st</sup> century CE. The ship carried on board another 240 cm-long iron anchor that was probably  
395 stored in the hold, as well as three additional wooden anchors found close to the bow, whose lead stocks  
396 range from 160.5 to 170 cm length and weigh between 200 and 250 kg (Liou and Domergue, 1990).  
397 Even though the excavation data regarding this shipwreck is insubstantial, the presence of a 350 cm-  
398 long iron anchor reveals that these large iron anchors were not specific to magnificent ships, such as the  
399 *Syracusia*, provided with eight iron anchors (Athenaeus, *Deipnosophistae* 5.208e) (Nowacki, 2002;  
400 Pomey and Tchernia, 2006; Castagnino Berlinghieri, 2010; Nantet, 2020b). Certainly, big anchors are  
401 expected to fit large merchantmen. Nonetheless, the Sud-Lavezzi 2 shipwreck reveals that even small  
402 ships could be fitted with such large anchors. As for the four iron anchors found in Marritza, the length  
403 of their shanks was respectively 270 cm, 290 cm, 310 cm, and 350 cm (Pallarés, 1986). The rope was  
404 still tied to their ring (Votruba, 2014), but the fact that they were not provided with a stock could indicate  
405 that they were stored onboard when the ship sank sometime between the end of the 1<sup>st</sup> and the middle  
406 of the 2<sup>nd</sup> century CE (Pallarés, 1986). The same reason would explain the absence of stock on the iron  
407 anchor found in Cabrera 4 shipwreck that is entirely preserved, which was loaded with lead ingots and

408 700 amphoras and dates to the early 1<sup>st</sup> century CE (Veny, 1979; Pons et al., 2001; Domergue et al.,  
409 2013). This anchor was 325 cm long with 180 cm arms-beam and was provided with a ring of 60 cm  
410 diameter. On the same shipwreck, the lower part of an iron shank (171 cm long) was also discovered.  
411 The absence of any hull does not allow us to draw conclusions on how sizable the ships outfitted with  
412 such large iron anchors would have been. The very low number of shipwrecks corresponding to large  
413 merchantmen that have been excavated makes it difficult to arrive at a definite answer, as no anchors  
414 have been found thus far from the few known examples, namely in La Madrague de Giens (75–60 BCE)  
415 (Tchernia et al., 1978; Pomey, 1982; Hesnard, 2012), Bou-Ferrer (1<sup>st</sup> cent. CE) (Juan Fuentes, 2018),  
416 and Caesarea (1<sup>st</sup> cent. CE) (Fitzgerald, 1994; Nantet, 2020a).

417  
418 The technology required to manufacture these remarkable anchors in antiquity is particularly  
419 noteworthy. Speziale, who investigated the iron anchor found in Nemi, noticed that they would have  
420 necessitated advanced forging skills (Speziale, 1931; Votruba, 2014). The weld-bulge on the stem of a  
421 few anchors displays the precise location of the joint where the two parts meet, resulting from the crown-  
422 to-shank smithing process that consisted of working on the two separate pieces before the final  
423 assemblage (Votruba, 2014). The lower part of the shank found in Lake Nemi includes a similar bulge  
424 whose purpose would also have been to avoid the slippage of the shank inside the wooden sheathing  
425 (Sadania, 2017). The breaking points of both shanks discovered in Aigues-Mortes Bay are located  
426 slightly below their center, thus revealing a weak point due perhaps by the montage of the two iron  
427 portions, or a weld-bulge, that was not preserved.

428  
429 The ring is a particular kind of element that is closely associated with the anchor. Subjected to all sorts  
430 of forces, it can be replaced (Sadania, 2017). Thus, this element can be of a much more recent date than  
431 the anchor to which it is attached. In any case, such heavy anchors as those found in La Grande-Motte  
432 probably would have required substantial cranes and devices for their lifting and handling. As the upper  
433 part of the hull is rarely preserved, the handling of such large specimens remains undocumented so far.

434  
435 The two anchor finds may indicate that a ship sank at that location, although current investigations have  
436 not revealed any wreck. Following Kapitän's suggestion for the anchor found in Qawra Point (Maritime  
437 Museum, Vittoriosa, Malta), those from La Grande-Motte also may have served as stationary weights  
438 (Zammit, 1964; Kapitän, 1978; Frost 1982; Kapitän, 1984; Purpura, 2003; Azzopardi et al., 2012;  
439 Votruba, 2014; Sadania, 2017). But it seems unlikely to use iron anchors that were so complicated to  
440 manufacture and therefore not economical for a permanent anchorage site (Haldane, 1986a). It seems  
441 more likely that the anchors were lost. In any case, the presence of these two large anchors would  
442 indicate a mooring area established in relation to one of the numerous channels that led from the sea to  
443 the lagoons and marshes located along the coast that extends westward from the Rhône delta. The latter  
444 were too shallow to let in large merchantmen. In the imperial period, it was usual for big ships to anchor

445 at the entrance of channels, where their cargo would have been unloaded onto smaller vessels (Dionysius  
446 3.44.3), like the *caudicariae* in Ostia (Boetto, 2008, 2016) or perhaps the example found in Mandirac  
447 (Jézégou et al., 2015).

448  
449 The most remarkable feature of the ring analysis is the pitch and fibers that remain under a concretion.  
450 Pitch is obtained from trees of the Pinaceae family after heating, and molecular analysis rules out the  
451 possibility of the use of bitumen. As for the plant fibers, they have been identified as Cyperaceae  
452 (*Cyperus canus*) or sedge family, which are hydrophilic herbaceous reeds, known for their fibrous  
453 properties, growing in wetlands in many parts of the world (clods, rivers and swamps, among other  
454 environments). They possess high levels of salt tolerance and are therefore well suited to be used as  
455 ropes on a ship. On the wreck of Ma'agan Mikhael (ca. 400 BCE), remnants of rope elements of the  
456 Cyperaceae family (*Scirpus holoschoenus*) were found attached to the crown, the shank of the anchor,  
457 and elsewhere on the ship (Charlton, 2003). These findings lead us to propose that what we found on La  
458 Grande-Motte anchor constitutes a puddening. This practice of the wrapping of tarred fabrics bound  
459 together with smaller ropes around the ring of an iron anchor was done in order to protect the cable from  
460 chafing. It was still in use on modern sailing ships (Aubin, 1702; Martelli, 1838), which were provided  
461 with hemp cables until the advent of chains in the early 19<sup>th</sup> century (Harland, 2013). So far, few anchors  
462 have shown any signs of puddening, with two notable examples being the anchors of the *Mary Rose*  
463 wrecked in 1545 (Votruba, 2014). Another particularly well-preserved example belonged to the HMS  
464 *Dictator*, which had been commissioned in 1783 and broken up in 1817, evidences a ring overlaid with  
465 a rope covered with canvas that had been pitched (Schwartz and Green, 1962). The anchor of the *Sydney*  
466 *Cove* merchantman, lost in 1797, also showed a four-stranded laid rope, made of coir fiber or kayar,  
467 which was built up from the short fibers of the outer husks of coconuts (Nash, 2002). Before our  
468 discovery, this practice had not been evidenced yet for ancient anchors. An example of this is the study  
469 of the rope that tied the Roman anchor of Maritza that did not detail whether it had been once connected  
470 to the ring (Pallarés, 1986).

471

## 472 6 Conclusion

473

474 The two largest finds evidence that imperial vessels moored in this bay. Our investigation has shown  
475 that without obvious organic material, isolated iron anchors can be dated and are able to provide valuable  
476 information about their manufacturing process. Radiocarbon analysis of the ring has provided a secure  
477 range of dates that does not rely on a typology. A closer examination of the ring reveals the presence of  
478 Cyperaceae plants that we have identified as the remains of small ropes that protected the cable from  
479 chafing. The analyses we have carried out on the ring are even more significant and necessary as the  
480 two iron anchors are the largest evidenced thus far for antiquity. For this reason, future investigation  
481 should be extended to additional anchors of various dimensions, not only those found in shipwrecks but

482 also isolated finds. These would help to determine if several details underlined in the present study,  
483 namely the puddening, are specific and particular to these large devices or whether investigations of  
484 smaller examples could also reveal their presence. Our team's findings suggest that this method can  
485 complement chrono-typological and archaeo-metallurgical studies to date anchors out of archaeological  
486 context. There is great benefit to a close examination of the ring with combined techniques that offers  
487 promising perspectives for revealing anchors' manufacturing in antiquity that hitherto have not attracted  
488 sufficient attention.

489

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501

### 502 **List of the figures**

503 Figure 1: Location of the town of La Grande-Motte on the Aigues-Mortes Bay with the rivers and the  
504 principal ancient settlements located in the Roman province of Narbonensis (above) and the modern  
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507 Figure 2: Plan of the excavation probes indicating the position of the anchors located at a 9 m depth and  
508 under 0.7 m of sediment (Drawing: M. Guérout, CAD: S. Berthaut-Clarac).

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512 Figure 6 a): The collected anchor ring and visualization of the state of the metal corrosion at both cut  
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514 the ring.

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517 related to the smelting process are evidenced. b) The identified chemical signatures are reported in the

518 cross-section of the sample. The signatures are not mixed within the sample and each appears to be  
519 associated with a specific metal piece.

520 Figure 8: a) A fiber fragment observed by SEM: long cells of sinuous walls, simple pits are visible, b)  
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523 Figure 9: Gas chromatogram (GC-MS, EI, 70 eV) showing the distribution of lipid biomarkers from the  
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525 analyzed as methyl esters and alcohols as acetates.

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527

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531 concretions

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535 Table S1 (supplementary material): Slag inclusion composition for detected compounds (weighted  
536 content). Contents normalized at 100%. S, Cl, Ti, V, Cr, Mn oxides were quantified but not presented  
537 in the table because their respective contents are always lower than 0.5%.

538

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**Table 1:** Dimensions in cm of anchor 1 (C1/1, C1/2, C2) and anchor 2 (C3/1, C3/2), their preserved fragments, and reconstructed measurements, the latter of which are estimated due to the presence of concretions

Dimensions (in cm)	Anchor 1			Anchor 2		
	C1/1 + C1/2 Preserved	C2 Preserved	<b>Overall C1+C2 Reconstructed</b>	C3/1 Preserved	C3/2 Preserved	<b>Overall C3 Reconstructed</b>
Length of shank	258	117	<b>375</b>	254	116	<b>370</b>
Length of stock	293	-	<b>293</b>	228	-	<b>228</b>
Length of arms	-	112 (right arm) 116 (left arm)	<b>112 (right arm) 116 (left arm)</b>	-	108 (left arm)	<b>108 (left arm)</b>
Arm-beams		185	<b>185</b>	-	180	<b>180</b>
Diameter of cable-ring (outer/inner)	32/24	-	<b>32/24</b>	35/23	-	<b>35/23</b>

**Table 2:** Radiocarbon dating results for the ring of anchor C3

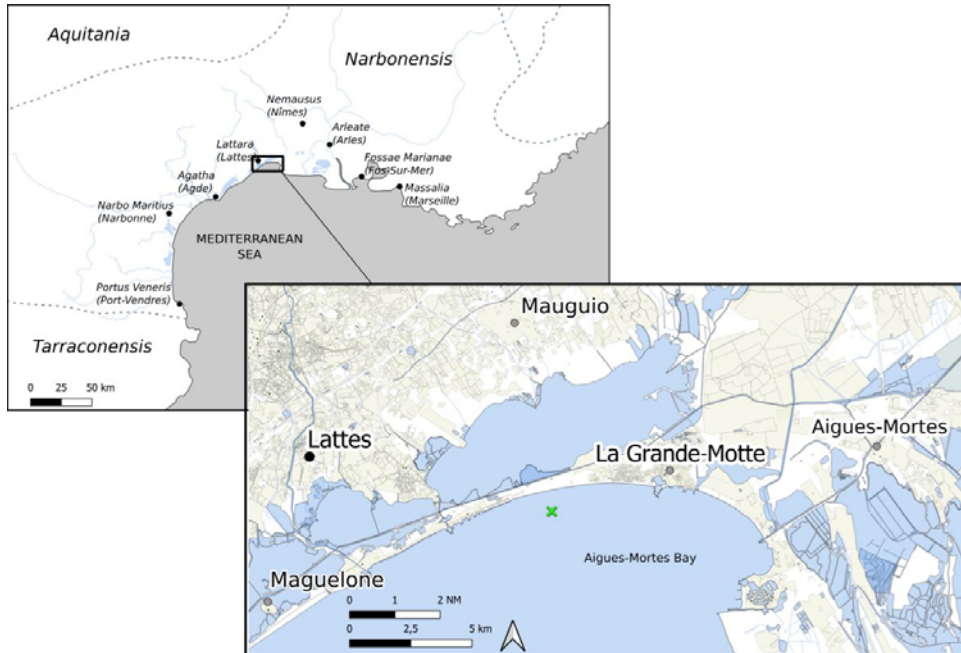
<b>Sample name</b>	<b>Lab.ID</b>	<b>Extracted carbon content (mg)</b>	<b><math>\delta^{13}\text{C}</math> (‰)</b>	<b>Radiocarbon age (BP, 1 <math>\sigma</math>)</b>	<b>Calibrated age (2<math>\sigma</math>, 95.4%)</b>
GDM20-org-1a	SacA 62202	0.28	-31.2	1940 $\pm$ 30	10 CE–204 CE
GDM20-org-1b	SacA 62203	0.11	-31.4	1960 $\pm$ 50	46 BCE–206 CE

**Table 3:** Anatomical characters observed with SEM

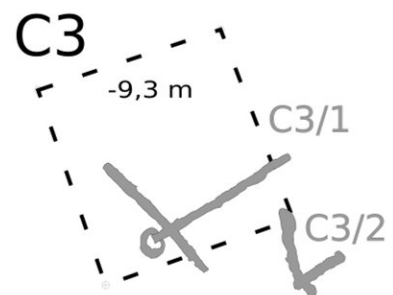
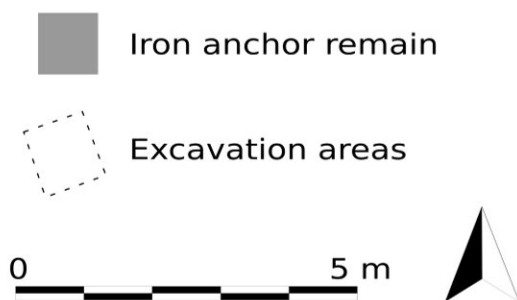
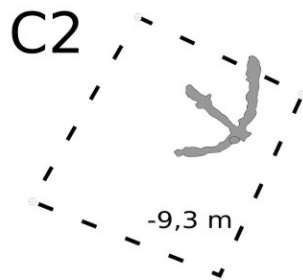
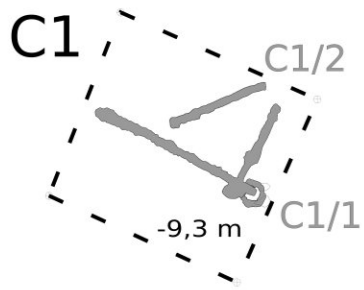
SEM Picture	Long cells	Short cells	Cell walls	Pits	Stomata	Phytoliths
MEB-02	Rectangular	Round	Sinuuous	Round	Paracytic	Saddle
MEB-03	Rectangular	-	Sinuuous	Simple 5 µm	-	Bride-shaped
MEB-04	-	-	Sinuuous	-	-	Saddle and bridge-shaped
MEB-05	-	-	-	-	-	Bride-shaped

**Table 4:** Radiocarbon dating results for the fibers of anchor C3

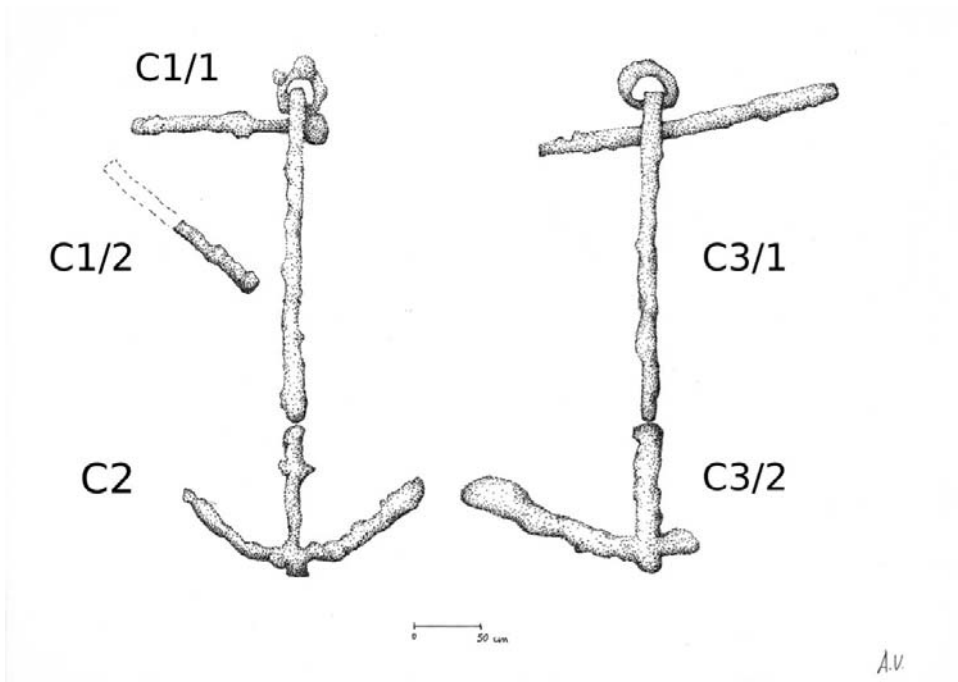
<b>Sample name</b>	<b>Lab. ID</b>	<b><math>\delta^{13}\text{C}</math> (‰)</b>	<b>Radiocarbon age (BP, <math>1\sigma</math>)</b>	<b>Calibrated age (<math>2\sigma</math>, 95.4%)</b>
<b>GDM20-org- fibers</b>	<b>SacA 64799</b>	<b>-22.5</b>	<b><math>1840 \pm 30</math></b>	<b>124 CE (91.8%) 250 CE 295 CE (3.6%) 310 CE</b>



**Figure 1:** Location of the town of La Grande-Motte on the Aigues-Mortes Bay with the rivers and the principal ancient settlements located in the Roman province of Narbonensis (above) and the modern towns nearby (below). The green cross indicates the find site of the anchors (CAD: S. Berthaut-Clarac. Base map and data from OpenStreetMap and OpenStreetMap Foundation).



**Figure 2:** Plan of the excavation probes indicating the position of the anchors located at a 9 m depth and under 0.7 m of sediment (Drawing: M. Guérout, CAD: S. Berthaut-Clarac).



**Figure 3:** Reconstruction of anchors 1 and 2 (Drawing: A. Verra).



**Figure 4:** Picture of the sampled ring (Photo: Author).

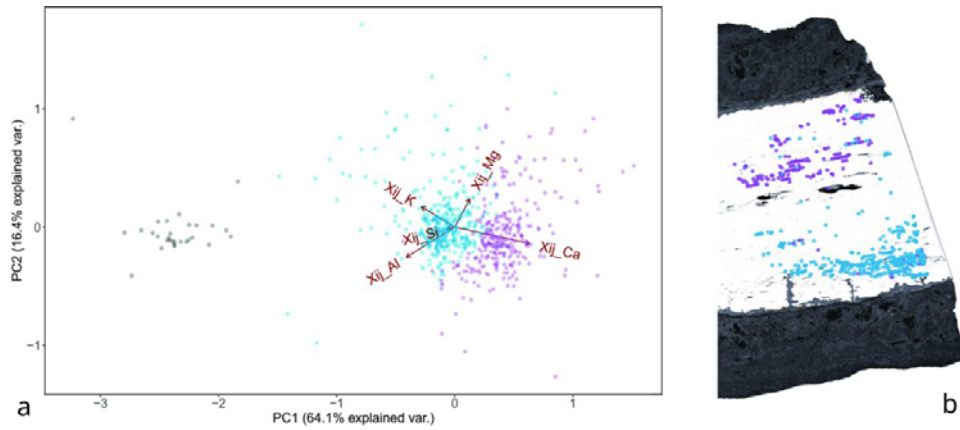




**Figure 5:** Detection of small fibers located on the puddening of the ring (Photo: Author).

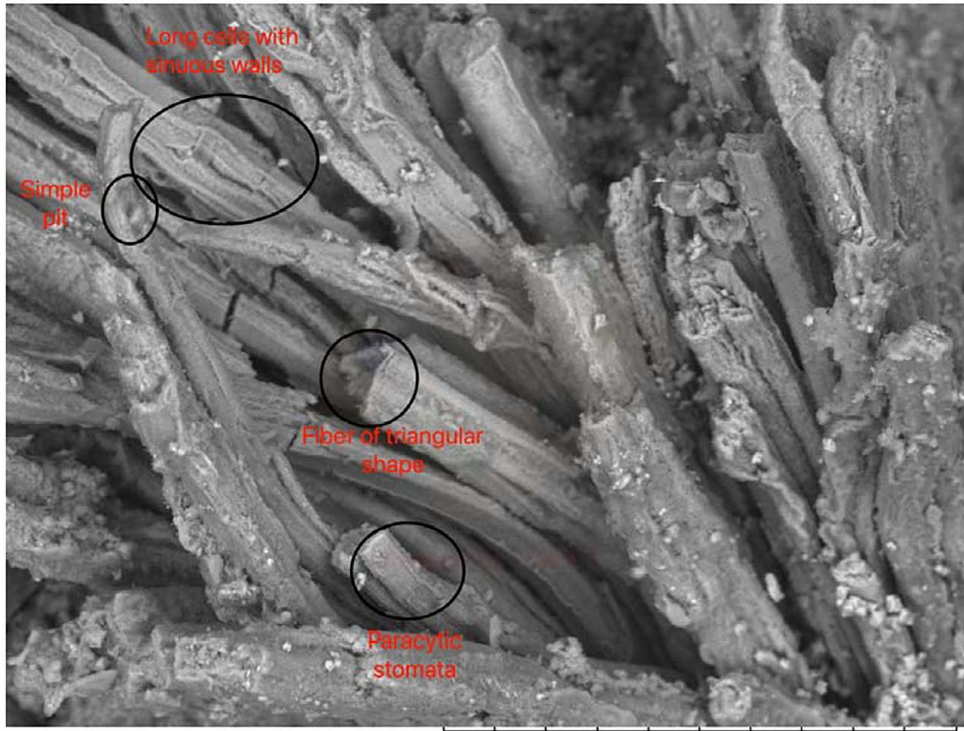


**Figure 6 a):** The collected anchor ring and visualization of the state of the metal corrosion at both cut ends. **b):** Cross-section of the sample and metallographic observation that highlight the ferritic nature of the ring.



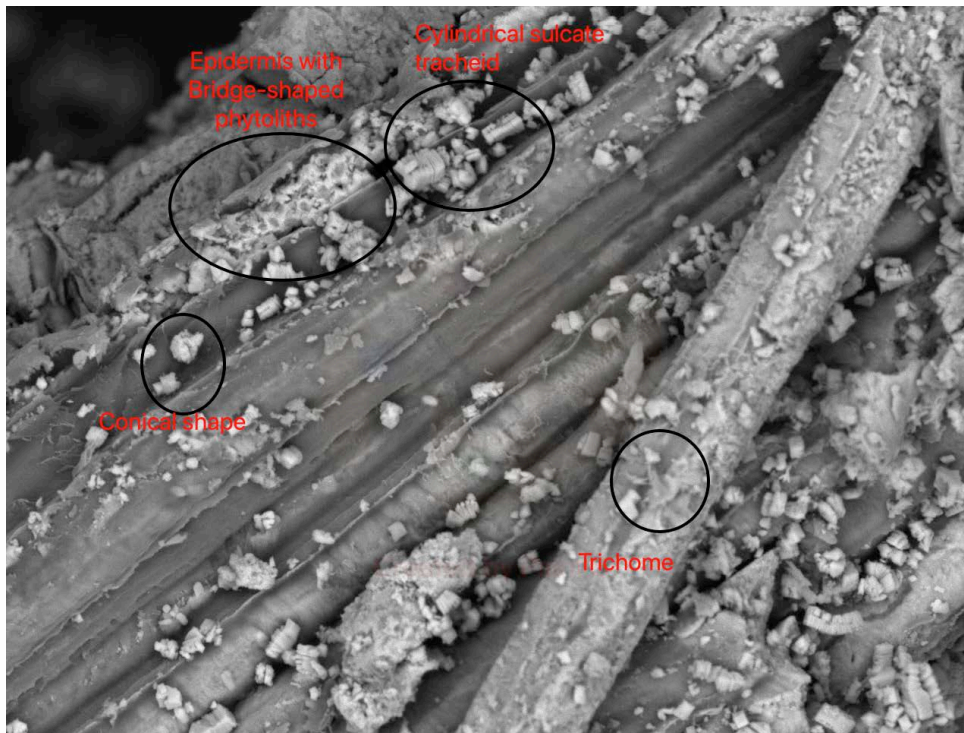
**Figure 7:** Treatment of SI chemical data on the sample. a) Multivariate analysis of the compositional data—ratios of elements  $X_{ij}$ - of the SI entrapped in the metal. Two chemical signatures (blue and violet) related to the smelting process are evidenced. b) The identified chemical signatures are reported in the cross-section of the sample. The signatures are not mixed within the sample and each appears to be associated with a specific metal piece.

**a**



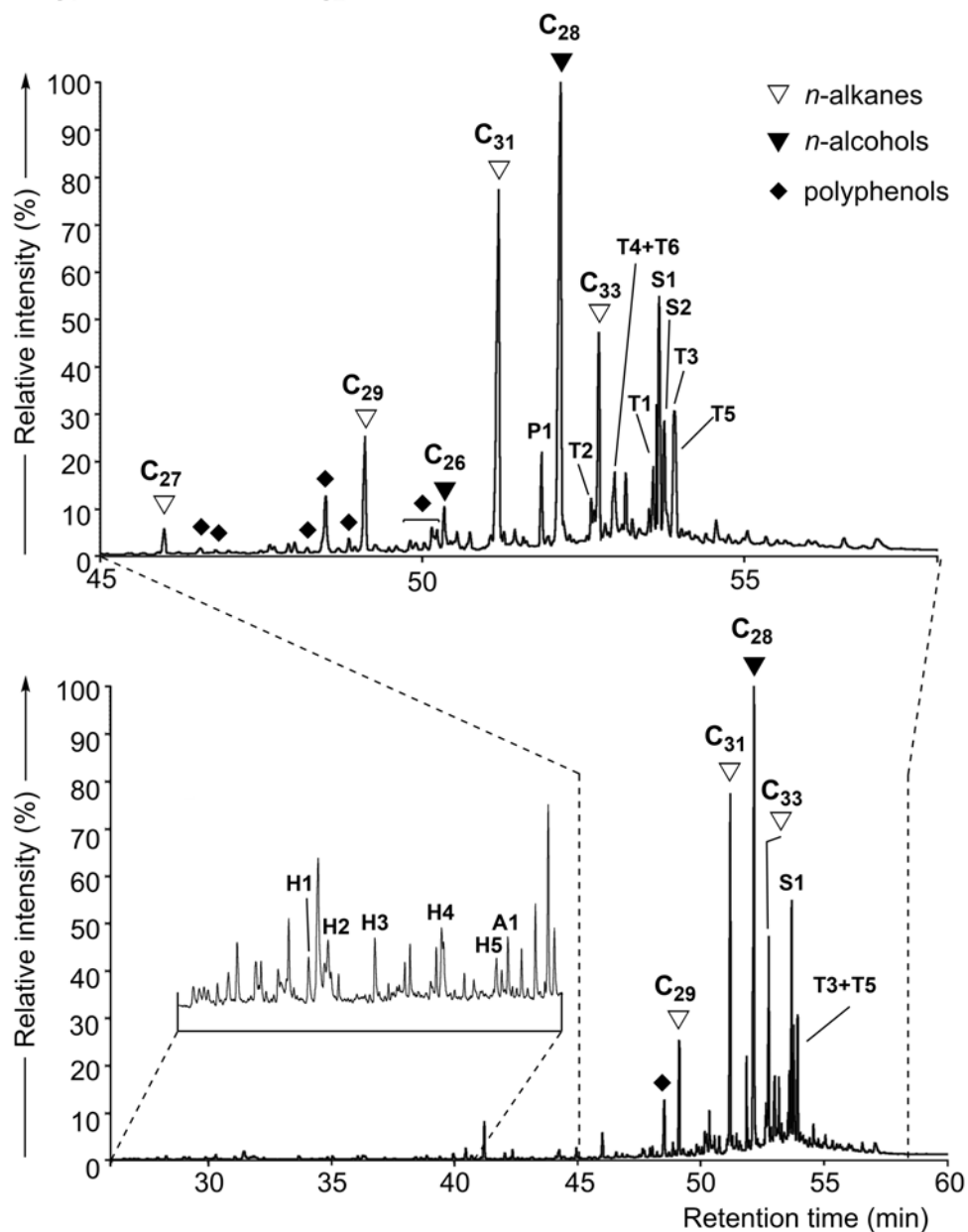
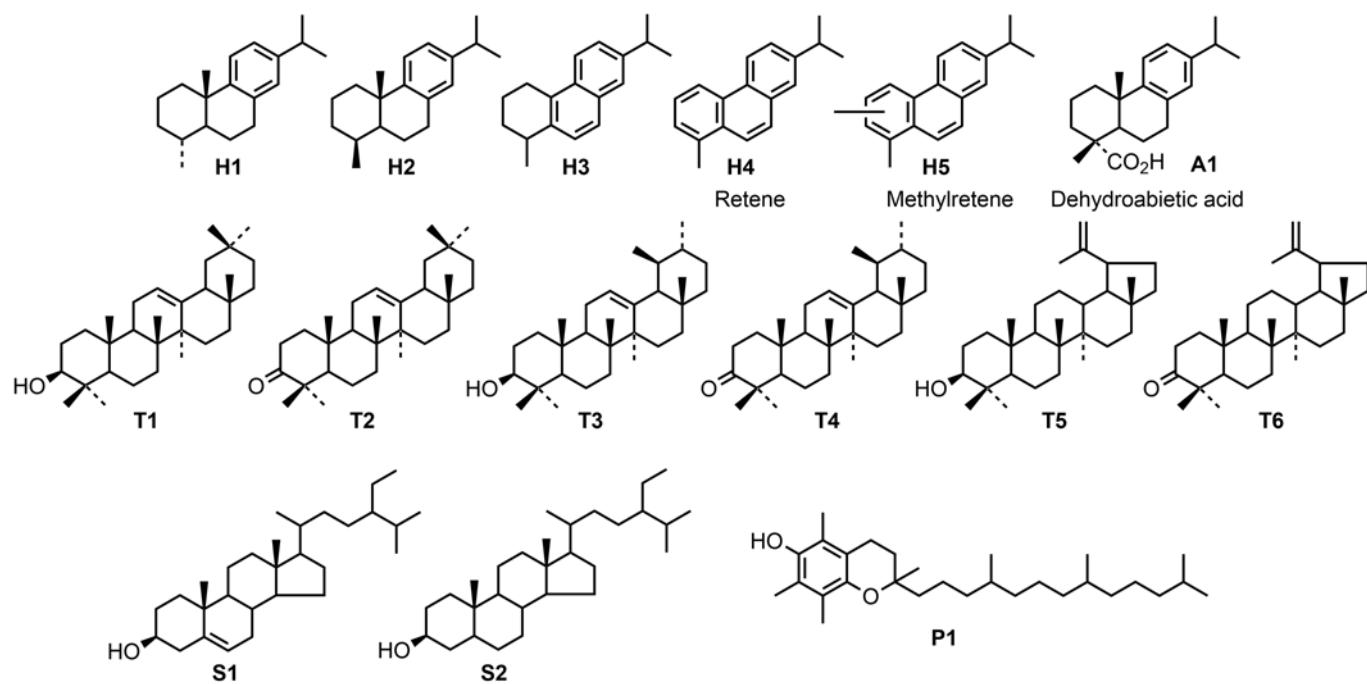
2021/05/06 A D7.2 x1.0k 100 um

**b**

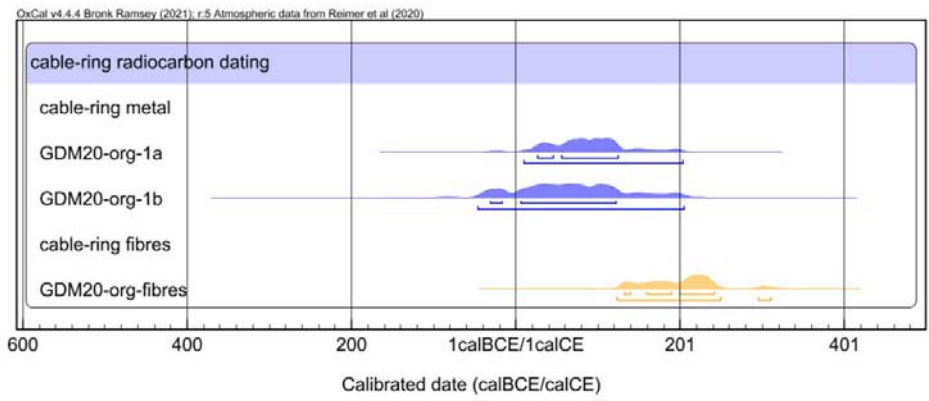


2021/05/06 A D7.9 x1.8k 50 um

**Figure 8:** **a)** A fiber fragment observed by SEM: long cells of sinuous walls, simple pits are visible, **b)** the ridge-shaped (or cylindrical sulcate tracheids) phytoliths are visible all over the surface of the plant remains (SEM photo: Author, Quai Branly-Jacques Chirac Museum).



**Figure 9:** Gas chromatogram (GC-MS, EI, 70 eV) showing the distribution of lipid biomarkers from the organic extract of a sample collected within the concretion covering the anchor ring. Acids were analyzed as methyl esters and alcohols as acetates.



**Figure 10:** Graphical representation of the radiocarbon dating of iron and fibers