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#### Embalmed heads of the Celtic Iron Age in South of France

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#### 23 Abstract

24 Ancient texts described that one of the most impressive ritual practices of the Celts during the

25 Iron Age was to remove the heads of enemies killed in battle and to display them, once 26 embalmed, in front of their own dwellings. An archaeological settlement excavation site in Le 27 Cailar, in southern France, has revealed a considerable number of examples of this practice, 28 known for many years thanks to literary sources and archaeological data: iconographic 29 representation of heads and human bones. Weapons were also exhibited alongside the severed 30 heads. Here we report the results of chemical investigations for the characterization of 31 embalm biomarkers liable to be present in eleven fragments of these human cranial remains. 32 These results may lead to answers to some of the archaeometric questions related to the subject of embalming in 3<sup>rd</sup> century BC Transalpine Gaul, mentioned by Greek authors, thus 33 34 advancing the knowledge of these ritual practices as part of the wider research into the proto-

35 historic societies of the Mediterranean coastal region.

### 36 Keywords

37 Iron Age, Celts, embalming, severed head, chemical analyses

38

### 39 **1. Introduction**

In the 3<sup>rd</sup> century BC, the number of wars and battles seems to increase in almost the whole of Western Europe. Indeed, hundreds of weapons have been found in sanctuaries and sacred places since they weren't display there before. In many of that places, human remains have been discovered with metal artifacts, and also fauna remains linked to sacrifice of animals (Buchsenschutz 2017; Brunaux, 2004; Barral et al., 2006). We know thanks to classical literary sources that at the end of battles, the Celts cut off their enemies' heads on the battlefield and carry them back to their settlements, hanging the decapitated heads on their 47 horse's neck. This very accurate picture of this practice is known through two fragments of ancient texts, written in the 1<sup>st</sup> century BC respectively by Strabo and by Diodorus of Sicily, 48 49 both recording the testimony of an ancient Greek, named Poseidonios, who travelled in the 50 south of Gaul around 100 BC (Strabo, IV, 4, 5 in Lasserre 1966). Other classical texts mentioned that fact (Polybius, Livy, ...) and many archaeological data illustrate this practice 51 52 too (Ciesielski et al. 2011; Armit 2012; Boulestin Henry Gambier 2012). At Entremont, an 53 Iron Age settlement in Provence, many pieces of sculpture have been discovered during one of the first dig in the South of France, showing decapitated heads, with one particular 54 55 sculpture representing a warrior on his horse, weapons (a sword and a spear) at his side, and a 56 severed head suspended from the horse's neck (Arcelin 2011), just as testified literary sources. Human bones were also discovered in many settlements belonging to the Second Iron Age 57 58 (Roquepertuse, Glanum, ...) and other sculptures and engraved stone too, in the whole South of France (fig. 1). In some places, archaeologists found human skulls with iron nails inside 59 them and in other places they found pillar or lintel with cavities head shaped (fig. 1). 60



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63

81

Fig 1. Map of discoveries in the South of France: human bones and sculptures. COLOR

64 Thus, displaying severed heads was a well-known practice, but it had never been observed in 65 recent digs in the South of France. That's why the discovery in Le Cailar is of great importance and provides us with a significant amount of new data (Roure et al. 2006), and the 66 opportunity to make new analysis, especially chemical ones to find if the heads have been 67 68 prepared as the Greeks testified it. Indeed, Strabo and Diodorus both wrote that the Celts 69 embalmed the heads, and they indicate both with 'cedar oil'; however, it could had been a 70 local Pinacea oil that Greeks named 'cedar' because the smell was close. That's why this paper's study aimed at verifying the presence of possible embalming remnants in 71 72 archaeological cranial fragments from Le Cailar. Chemical analyses using GC-MS were so performed in order to characterise organic components liable to be present in eleven from 73 74 these human cranial fragments

Embalming and other mummification phenomena are well-documented worldwide with much
of both the scientific and the archaeo-historic academic literature documenting the best
surviving examples of embalming in pharaonic dynasties of Egypt (Łucejko et al., 2012;
Ménager et al., 2014; Nicholson and Shaw, 2000), and mummification has also been proved
in Bronze Age in Britain (Parker Pearson et al. 2005). Our paper will present another example
of that kind of practice.

- 82 **2.** Experimental section
- 83 **2.1. Sample description**

84 The Iron Age settlement of Le Cailar was situated near a wide lagoon connected to the Rhône River. It was occupied from the 6<sup>th</sup> century BC until the Roman period in Gaul (1<sup>st</sup> century 85 AD). The fortified settlement was located on a small hill and was also a harbour for 86 87 Mediterranean traders (Etruscans and Greeks at that time). Near the fortification, and 88 probably close to one of the gates, a large public area where inhabitants exposed weapons and 89 skulls was discovered, displayed on a large space (fig. 2). The archaeological context is very 90 clear: there is no doubt it is an open area where the heads and weapons were displayed. We 91 have stratigraphical and chronological evidence for each level with display of bones and 92 metal artifacts (Roure et al. 2017). Remnants of metal (mainly weapons), potteries and animal 93 bones, were intermingled with the human skulls. Both ceramic and metallic remains allowed a 94 precise chronology of this embalming event to be obtained and forty Massilian coins were 95 also found in the same place, which again confirms the chronology of the 3<sup>rd</sup> century BC (that's why there is no radiocarbon dating). Amphora and vessels from the Greek city of 96 97 Massalia and vases from Italy and Spain all belong to this period and the metal artifacts are all 98 linked to the latenian typology (La Tène B2-C1). The stratigraphy and artifacts showed that 99 several deposits happened: the first deposits corresponded to the end of the 4<sup>th</sup> century or to the very beginning of the 3<sup>rd</sup> century BC, and that the trophy heads and weapons were 100 displayed throughout the 3<sup>rd</sup> century BC until *circa* 200 BC, when the area was covered by 101



- 102 mud.
- 103

104	Fig 2.: Maps of Le Cailar excavation with the distribution of each kind of remains and localization of samples
105	(on the left) and only the metal artifacts and the human bones (on the right).
106	(all the deposit mapped together) COLOR
107	

About 2700 fragments of human bones were recorded during ten excavation campaigns: all the fragments are part of the skull, except for six little pieces of cervical vertebrae (fig.3). Many of the skulls presents cut marks, linked not only to the act of decapitation, but also to some act connected to preparing the heads for display: removal of cervical vertebrae and aperture of the postero-inferior portion of the cranium, probably carried out in order to

- 113 remove the brain; and tongue ablation, or at least the scraping of the muscles under the
- 114 mandible (Ciesielski et al. 2014).

Identified Pieces	NR
skull	1078
skull/face	2
face	286
mandible	117
teeth	973
cervical vertebrae	4
skull pieces non identified precisely	216
Total	2676



global MNI = 51

	Number of remains	Weight (g)	median weight (g)	mean weight (g)
skull	1078	12480,3	8,3	16
skull/face	2	100,3	-	-
face	286	2205,3	6,5	9
mandible	117	2997,9	20,1	28,1
cervical vertebrae	4	14,2	2,5	3,6
Total	1487	17798	37,4	56,7

- 115
- 116
- Fig 3. : table of human remains and map of cranial NMI from Le Cailar (2003-2011) COLOR

The discovery of the skulls was immediately related to the above-mentioned ancient texts from Strabo and Diodorus. These texts indicated that the Gauls "embalmed the head of the most famous enemy with cedar oil" (Strabo, IV, 4, 5 *in* Lasserre 1966), and this explains why chemical analysis, as opposed to other types of analyses, was undertaken. Preliminary chemical analyses on human bones were carried out in the search for any traces of biological products which could have been used to prepare these heads for their display, even though no macroscopic or microscopic remains were visible.

124 Eleven cranial fragments were selected (Table 1, Fig. 4), from each of which two powder 125 samples (100 mg to 150 mg each) were taken, first from the exterior and then from the 126 interior surfaces. A total of twenty-two samples were analyzed. Fragments were chosen from 127 the skulls: frontal, zygomatic, parietal, originating from different deposits. At least, our panel would be representative, even though the way the 11 pieces were chosen from among 2800 128 129 was random, because there wasn't any visible residue. All the human remains were precisely 130 recorded with three coordinates (x, y, z) and registered by number (except the pieces 131 discovered at the beginning of the excavation and before the installation of this protocol).



133 Fig 4. Pictures of a. Total assemblage b. CLR K16 R9 286 exterior surface c. CLR N17 R3 53 interior surface 134 cranial fragments (after Ghezal and Gosnell). COLOR



Alpha-numeric context number	General bone identification
CLR 07	Mandible (central section)
CLR 04 X5	Mandible (right side)
CLR 03 X103	Mandible (left side)
CLR 04 X11	Parietal
CLR 03 X29	Parietal
CLR 03 X44	Parietal
CLR M18 R6 570	Frontal
CLR N17 R0 8	Frontal
CLR K15 R5 340	Frontal
CLR K16 R9 286	Zygomatic (left)
CLR N17 R3 53	Zygomatic (left)

Five remains of fauna (Table 2), discovered strictly in the same place and at the same level as 136 the human bones, were also analysed using identical protocols, in order to verify if the 137

products found could come from taphonomic bias or if they could be link to a specific 138 139 practice for the human heads.

140

Alpha-numeric context number
K15 R5 361
K15 R5 354
K16 R9 304
M18 R6 535 A
M18 R6 538

# 142 **2.2. Materials**

143

144 Dichloromethane was of GC grade and purchased from Merck (Darmstadt, Germany). High 145 purity water (18.3 M $\Omega$ .cm) was obtained from a Milli-Q purification system (Millipore).

146 Derivatisation was made using BSTFA (N,O-bis(trimethylsilyl) trifluoroacetamide) with 1 % 147 TMC (trimethylchlorosilano) purchased from Sigma Aldritch

147 TMC (trimethylchlorosilane) purchased from Sigma Aldritch.

148 149

# 150 **2.3. GC-MS analyses**

151

152 Each powdered cranial sample was added to 6 mL of dichloromethane and sonicated (2 min, 153 70% amplitude) using an ultrasonic probe (Vibra-Cell model 75186). This volume was 154 necessary in order to obtain the immersion of the probe in the extraction solvent. The samples 155 were centrifuged (30 min, 6000 rpm). The supernatant corresponding to the organic extract was 156 split into two, in order to perform GC-MS analyses.

157

158 Before the GC-MS analyses, the organic extracts were derivatised by trimethylsilylation. For

- 159 this purpose, the solutions were evaporated to dryness under nitrogen and mixed with  $100 \ \mu L$
- 160 of BSTFA with 1 % TMC for 30 min at 70°C. The trimethylsilylated extracts were dried under

161 nitrogen and dissolved in 500  $\mu$ L of hexane. All the samples were filtered through a 0.45  $\mu$ m

162 PTFE filter before injection.

163 GC-MS analyses were carried out applying a Thermo Scientific<sup>TM</sup> Focus gas chromatographic

164 system mounted with a Thermo Scientific Al 3000 auto-injector and coupled with a  $ITQ^{TM}$  700

165 Series GC-Ion Trap Mass Spectrometer (Thermo Fisher Scientific Inc.). GC separation was

166 performed on a fused silica capillary column TG-5MS (Thermo Fisher Scientific, alvc), with a 167 stationary phase (5% diphenyl 95% dimethyl polysilovane phase)

167 stationary phase (5% diphenyl-95% dimethyl-polysiloxane phase).

168 A volume of 1  $\mu$ L for each sample was injected in splitless mode and the injector temperature

169 was set at 250°C. Molecular components were eluted using helium at a constant flow of 1.2

170 mL/min. The following temperature programme was used: initial temperature 50  $^\circ$ C for 2 min,

171 50 to 220 °C at 8 °C/min, 220 to 260 °C at 2 °C/min and 260 to 330 °C at 10 °C/min.

172 The mass spectra were recorded in Electron Impact mode with an electron ionization voltage of

173 70 eV, an ionisation time of 25,000  $\mu$ s and a mass range of 50 to 650 *m/z*. The ion trap and

174 interface transfer line were respectively at 250 °C and 300 °C.

175 Thermo Xcalibur<sup>TM</sup> 2.2 software (Thermo Fisher Scientific Inc.) was used for instrumental 176 control and data acquisitions.

177 Mass spectra peak assignment was based on a comparison with internal mass spectrum 178 database (from commercial standards and from fresh and artificially-aged resins and oils) and 179 NIST database (NIST MS Search 2.0).

180

## 181 **3.** Results and discussion

All of the components were identified according to their specific mass data (base and molecular ions), their retention times in comparison with standard molecules and the specialized literature (Table 3).

For an accurate interpretation of GC-MS results, the contribution of lipids from the bone cannot be disregarded. Fresh bones contain significant amounts of cholesterol and a lesser 187 concentration of fatty acids associated with marrow (Colonese et al., 2015; Evershed et al., 188 1995). Previous analyses of archaeological bones revealed only the presence of cholesterol, 189 together with its diagenetic degradation products, especially cholest-5-en-3B-ol-7-one (Collins 190 et al., 2002; Colonese et al., 2015; Evershed et al., 1995; Stott et al., 1997). However, traces 191 of saturated fatty acids (primarily  $C_{14:0}$ ,  $C_{16:0}$  and  $C_{18:0}$ ), a lesser concentration of oleic acid 192 ( $C_{18:1}$ ) and a low quantity of linoleic acid ( $C_{18:2}$ ) were recently detected in the analyses of 193 human bones for archeological purposes (Colonese et al., 2015).

The total ion current of the lipid extract from CLR K16 R9 286 sample is presented in Figure 5 and shows that almost all lipid extracts from the analysed bones exhibited the presence of saturated fatty acids C<sub>9:0</sub>, C<sub>14:0</sub>, C<sub>16:0</sub> and C<sub>18:0</sub>, monoacylglycerols, cholesterol and its degradation products: cholest-5-en-3 $\beta$ ,7 $\beta$ -diol and cholest-5-en-3 $\beta$ -ol-7-one.

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The fatty acid composition of the different samples is presented in Figure 6, showing a chromatogram of the internal CLR K16 R9 286 sample with extracted signals at m/z 117 and the base peak of main saturated fatty acids. The chromatogram shows that almost all lipid extracts from analyzed bones exhibited the presence of saturated fatty acids C<sub>9:0</sub>, C<sub>14:0</sub>, C<sub>16:0</sub> and C<sub>18:0</sub>. Some unsaturated fatty acids were also detected (C<sub>14:1</sub>, C<sub>16:1</sub>, C<sub>17:1</sub>, C<sub>18:1</sub>).

212



Fig 6. Gas chromatogram of internal CLR K16 R9 286 sample with extracted signals of *m/z* 117. Cn:x fatty acids with n carbon atoms and x unsaturations. All the fatty acids were detected in their trimethylsilylated form.

The high amount of saturated fatty acids, monoacylglycerol (MAG), glycerol, cholesterol and
its observed degradation products, are characteristic of degraded animal fats (Mottram et al.,
1999). The high ratio of palmitic acid compared with that of stearic acid, in addition to the
presence of β-sitosterol are indicative of possible plant-origin fats. However, the contribution
from endogenous bone fats cannot be discounted.

Unfortunately, it is not possible to assign a precise plant origin on the basis of fatty acids
alone using conventional chromatographic methods, because of the lipid thermal degradation
leading to changes in saturated fatty acid proportions (Nawar, 1969).

Six of the eleven human samples (CLR 03 X44; CLR M18 R6 570; CLR N17 R0 8; CLR K15 R5 340; CLR K16 R9 286; CLR N17 R3 53) contained diterpenoic compounds, degradation products of abietic acid and biomarkers of conifer resin. The gas chromatogram of the internal K15 R5 340 sample is presented in Figure 7 with extracted signals at m/z 239, m/z 253, m/z 219 and m/z 241 base peaks respectively of dehydroabietic acid, 7-oxo-dehydroabietic acid, retene, palustric acid (26.24 min) and isopimaric acid (26.66 min).

The dehydrogenation of abietic acid leads to dehydroabietic acid. This compound was the most abundant diterpenoid detected in our samples, followed by its oxidation product, 7-oxodehydroabietic acid. Retene is the final product of the thermal degradation of abietane skeleton diterpenoids. The detection of such aromatic compounds in these samples is characteristic of intense heating of the resin from the tree belonging to the Pinaceae family (Marchand-Geneste and Carpy, 2003).

The traces of linear *n*-alkanes ( $C_{23}$ - $C_{33}$ ) and *n*-alkanols ( $C_{12}$ - $C_{26}$ ) which were detected are more likely caused by soil contamination. In fact, these compounds were already detected in significant amounts in the soil (Poirier et al., 2005).



Fig 7 Gas chromatogram of internal CLR K15 R5 340 sample with extracted signals of *m/z* 239, 253, 219 and 241. Except for retene, all the terpenoid compounds were detected in their trimethylsilylated form.

243

244 Interestingly, in the lipid extracts from faunal samples only cholesterol was preserved (data 245 not shown). Fatty acids initially present in the bones seemed degraded and terpenoid 246 compounds were not detected. These results suggest that lipids observed in the human skull 247 extracts originate not only from human bones, but also from vegetal or animal fats and allowed to eliminate the hypothesis of external contaminations for all the detected substances 248 linear 249 excepted *n*-alkanes and *n*-alkanols.

	CLR 07		LR CLR 7 04 X5		CLR 03 X103		CLR		CLR 03		CLR 03		CLR		CLR		CLR		CLR		CLR	
							04	X11	2	X29	Х	X 44		M18		N17		15	K16		N17	
														6	R	08	R5 340		R9 286		R3 53	
													570									
	int	ex	in	ex	in	ext	in	ex	in	ext	in	ext	int	ext	in	ex	in	ex	in	ex	in	ext
		t	t	t	t		t	t	t		t				t	t	t	t	t	t	t	
Monocarboxylic acids																						
C <sub>8:0</sub> and less	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
C9:0	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
C <sub>10:0</sub>	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
C <sub>11:0</sub>	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-
C <sub>12:0</sub>	-	-	-	-	-	-	-	-	+	+	+	+	+	+	+	+	+	+	+	+	-	-
C <sub>13:0</sub>	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	-	+	+	-	-
$C_{14:1\Delta 9}$	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	-	-	-
C <sub>14:0</sub>	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
C <sub>15:0</sub>	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
Cis $C_{16:1 \Delta 9}$	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
C <sub>16:0</sub>	+	+	+	+	+	+	+	+	+	+	+	+	+	+	-	-	+	+	+	+	+	+
C <sub>17:1</sub>	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	-	+	-	-	-
C <sub>17:0</sub>	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	-	+	+	-	-
<i>Cis,Cis</i> C <sub>18:2 Δ 9,12</sub>	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	-	+	+	-	-
Cis C <sub>18:1 <math>\Delta</math> 9</sub>	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-
C <sub>18:0</sub>	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
C <sub>19:0</sub>	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
Monoacylglycerol																						
MAG C <sub>14:0</sub>	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
MAG C <sub>16:0</sub>	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
MAG C <sub>18:0</sub>	+	+	+	+	+	+	-	-	+	+	-	+	+	+	+	+	+	+	+	+	+	+
Steroidal compounds																						
Cholesterol	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+

+

-

+

+

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+

+

+

+

+

+

-

+

+

+

+

+

+

+

+

ß-Sitosterol

diol

Cholest-5-en-3β,7β-

Cholest-5-en-3ß-ol-7-

+

-

+

+

-

+

+

Table 3. Lipidic composition of organic extracts

+

-

+

+

+

+

+

+

+

one																						
Glycerol	-	-	+	-	+	+	-	-	+	-	+	+	-	-	+	+	-	-	-	-	+	+
n-Alkanes																						
C <sub>23</sub>	-	-	-	-	-	-	-	-	-	-	+	-	-	-	-	+	-	-	-	-	-	-
C <sub>24</sub>	-	-	-	-	-	-	-	-	-	-	+	-	-	-	-	-	+	+	+	+	-	-
C <sub>25</sub>	-	-	+	-	-	-	+	+	-	-	+	-	-	-	-	+	+	+	+	+	-	-
C <sub>26</sub>	-	-	+	-	-	-	+	+	-	-	+	-	-	-	-	+	+	+	+	+	-	-
C <sub>27</sub>	+	+	+	-	-	-	+	+	-	-	+	-	-	-	-	+	+	+	+	+	-	-
$C_{28}$	+	+	+	-	-	-	+	+	-	-	+	-	-	-	-	+	+	+	+	+	-	-
C <sub>29</sub>	+	+	+	-	-	-	+	+	-	-	+	-	+	+	-	+	+	+	+	+	-	-
C <sub>30</sub>	-	-	+	-	-	-	+	+	-	-	+	-	+	+	-	+	+	+	+	+	-	-
C <sub>31</sub>	+	+	+	-	-	-	+	+	-	-	+	-	+	+	-	-	+	+	+	+	-	-
C <sub>32</sub>	+	+	-	-	-	-	+	+	-	-	+	-	+	+	-	+	+	+	+	+	-	-
C <sub>33</sub>	+	+	-	-	-	-	-	-	-	-	+	-	+	+	-	-	-	-	-	-	-	-
n-alkan-1-ol																						
C12 OH	-	-	-	-	+	+	-	-	-	-	-	-	+	+	+	+	+	+	+	+	-	-
C14 OH	-	-	-	-	+	+	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
C15 OH	+	+	-	-	-	-	-	-	-	-	-	-	+	+	+	+	+	+	+	+	+	+
C16 OH	-	-	-	-	+	+	-	-	-	+	-	-	+	+	+	+	+	+	+	+	+	+
C17 OH	-	-	-	-	+	+	+	+	-	-	-	-	-	-	+	+	-	-	+	+	+	+
C18 OH	+	+	+	+	+	+	+	+	-	+	-	-	+	+	+	+	+	+	+	+	+	+
C19 OH	-	-	-	-	+	+	-	-	-	-	-	-	+	+	-	-	+	+	+	+	+	+
C20 OH	+	+	-	-	+	+	-	-	-	+	-	-	+	+	+	+	+	+	+	+	+	+
C21 OH	+	+	-	-	+	+	-	-	-	-	-	-	+	+	-	-	+	+	+	+	+	+
C22 OH	+	+	-	-	+	+	+	+	-	-	-	-	+	+	+	+	+	+	+	+	+	+
C23 OH	+	+	-	-	+	+	-	-	-	-	-	-	+	+	+	+	+	+	+	+	+	+
C24 OH	+	+	-	-	+	+	+	+	-	-	-	-	+	+	+	+	+	+	+	+	+	+
C25 OH	+	+	-	-	-	-	-	-	-	-	-	-	+	+	-	-	-	-	-	-	-	-
C26 OH	+	+	-	-	+	+	+	+	-	-	-	-	+	+	+	+	+	+	+	+	-	-
Diterpenoids																						
Palustric acid	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
Isopimaric acid	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-	+	+	+	+	-	-
DHA	-	-	-	-	-	-	-	-	+	-	+	+	+	+	+	+	+	+	+	+	-	-
7-Oxo-DHA	-	-	-	-	-	-	-	-	-	-	+	-	+	-	-	-	+	tr	tr	tr	-	-
Retene	-	-	-	-	-	-	-	-	-	-	+	-	-	+	-	-	+	-	-	-	-	-

Abbreviations: Cn:x, Monocarboxylic acid with n carbon atoms and x unsaturations; MAG Cn:x, Monoacylglycerols with n carbon atoms and x unsaturations; Cn, alkane with n carbon atoms, CnOH, alkan-1-ol with n carbon atoms; DHA, Dehydroabietic acid; +, presence; -, absence; tr, traces.

### 1 Conclusion

Chemical analyses using GC-MS were performed in order to characterise organic components
liable to be present in eleven human cranial fragments discovered at the Le Cailar
archaeological site in the south of France.

5 Thanks to this study, we demonstrated that some of the severed heads exhibiting by the Celts 6 were embalmed. We knew thanks to literary sources and to archaeology that the Celts 7 removed the heads of their enemies slain on the battlefield and that they exhibited them in 8 public spaces, maybe as an expression of the bravery and the strength of the community and 9 of its warriors (Boulestin Henry Gambier 2012, Ciesielski et al. 2011).

We speak of 'mumification' because Ancient Greek texts clearly assert that Celts used to embalmed the head with cedar-oil – or a local pinacea oil that Greeks named 'cedar' – and wanted to keep those heads for a long time. Moreover, Strabo and Diodorus wrote: "They never gave back the head belonging to the most famous and brave people, even for an equal weight of gold" (Strabo, IV, 4, 5 *in* Lasserre 1966). That sentence means it was possible to recognized the severed head.

16 In fact, analyses highlighted the presence of saturated and unsaturated fatty acids, 17 monoacylglycerols, sterols, alkanes, alkanols and biomarkers of conifer resins. Resins were 18 usually heated and mixed with plant oil, which may explain the presence of retene and the 19 high amount of fatty acids in these samples, notably palmitic and stearic acids. The use of a 20 mixture of resin and oil is documented in antiquity, in many societies and at different periods, 21 for their antibacterial, anti-oxidative and aromatic properties (Langenheim, 2003). Concerning 22 the Celts, the practical results of that kind of treatment – antiseptic and avoiding smells – are 23 maybe the first and main reason why they have done it, but this is linked to the will to 24 preserved the head. None of the fauna remains analyzed contained that kind of biological 25 things, so it is not something coming from the soil of this area but really a specific and voluntary practice of the Celtic people in order to embalmed the head. 26

27 The precise process of embalmment is quite difficult to know for that period: maybe the head 28 was dived in cedar-oil or the local pinacea oil; maybe the heads were just covered with the 29 pinacea mixture with some tool which totally disappeared with time. As noted above, 30 biological study of the bones remains has showed many cut marks, linked to preparing the 31 heads – probably tongue ablation and removing of the brain (Ciesielski et al. 2014). Anyway, 32 enough oil had to be used to penetrate inside the bone, but the process could be long: oil could 33 had penetrated slowly into the bone, while time was running, and sun and rain affecting the 34 heads exposed outside. It is also possible that Pinaceae oil was used several times, all along 35 the display, in order to preserve the head. In both case, that could explain why it is the 36 parietal/frontal where the Pinaceae is found, because they were the most visible and exposed 37 pieces.

As an important point arising from this study, it would be of great interest to determine when this specific practice actually began, in the early 3<sup>rd</sup> century BC or before, at the end of the 4<sup>th</sup> century BC. Further analyses should be carried out in order to answer this question. We will also have to question if the skulls came only from enemies or from ancestors at the same time (Ciesielski 2017), as it often happened in head hunting societies (Boulestin Henry Gambier 2012). Finally, we have to determine if the process was used for all the head or only some of them.

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- 8 **Conflict of interest:** The authors declare that they have no conflict of interest.
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