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A GRAY WHALE IN MAGDALENIAN PERIGORD.

SPECIES IDENTIFICATION OF A BONE PROJECTILE POINT FROM LA MADELEINE (DORDOGNE, FRANCE) USING COLLAGEN FINGERPRINTING

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The Cetacean bone artifact described in this paper is the result of excavations carried out in La Madeleine (Tursac, Dordogne) under the direction of D. Peyrony, between 1910-1913. It was discovered in the middle layer (“*couche moyenne*”) of the stratigraphy described by Capitan and Peyrony and subsequently attributed to the “Magdalenian V” by H. Breuil (i.e. the beginning of the Upper Magdalenian). This layer corresponds to layers C25 to C20 of the stratigraphy established later by J.-M. Bouvier. Two radiocarbon dates are currently available (C21: 12750 ± 240 BP, Ly 920; and C25: 13070 ± 190 BP, Ly 921). The artifact was recently identified as Cetacean bone during an integrated study of the bones and reindeer antlers kept at the Musée National de Préhistoire (Peyrony and Bouvier collections). It is a fully worked object, elongated in shape (129 X 13.2 X 12.8 mm; 18.6 g) with straight outline and profile, a circular cross-section and parallel sides. It is interpreted as a mesio-proximal fragment of projectile point. Collagen peptide mass fingerprinting (PMF) was applied to taxonomically identify the bone artefact, as a part of the PaleoCet project. Also known as ZooMS, collagen PMF discriminates taxonomic groups based on differences in the collagen protein sequence observed through Matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF) mass spectrometry. The bone subsamples from the exterior and interior of the artifact produced similar MALDI-TOF spectra, both of which could be confidently identified as gray whale (*Eschrichtius robustus*). The artifact from La Madeleine is the first evidence of the presence of this species in Atlantic waters at the end of MIS2, suggesting continuity between the Pleistocene and Holocene populations of this species. Moreover, to our knowledge, this gray whale bone artifact represents the oldest identified gray whale in an archaeological context, thus first witness of the millennia-old interactions between humans and this highly coastal whale species. The manufacturing of the La Madeleine artifact on the Basque coast, followed by its circulation through the western Pyrenees and then towards the north at the east of Landes and Gironde up to Périgord (a travel distance of roughly 350 km), might be the most plausible route taken by this artifact to reach La Madeleine.

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KEY-WORDS Bone tool, collagen peptide mass fingerprinting, Gray Whale (*Eschrichtius robustus*), La Madeleine, Magdalenian, projectile point.

**Une baleine grise dans le Périgord magdalénien :
identification taxinomique d'une pointe de
projectile en os de La Madeleine (Dordogne,
France) par empreinte peptidique de masse du
collagène.**

L'artefact en os de cétacé décrit dans cet article provient des fouilles effectuées entre 1910 et 1913 à La Madeleine (Tursac, Dordogne) sous la direction de D. Peyrony. Il a été découvert dans la couche moyenne de la stratigraphie décrite par Capitan et Peyrony («Magdalénien V» de H. Breuil, c'est-à-dire le début du Magdalénien supérieur), et qui correspond aux couches C25 à C20 de la stratigraphie établie ultérieurement par J.-M. Bouvier. Deux dates radiocarbone sont actuellement disponibles (C21: 12750 ± 240 BP, Ly 920 ; et C25: 13070 ± 190 BP, Ly 921). La pièce a récemment été identifiée comme os de cétacé lors d'une étude intégrée des os et bois de rennes conservés au Musée national de Préhistoire (collections Peyrony et Bouvier). Il s'agit d'un objet entièrement façonné, de forme allongée (longueur X largeur X épaisseur : 129 X 13,2 X 12,8 mm ; masse 18,6 g) avec un contour et un profil droits, une section circulaire et des bords parallèles. Il est interprété comme un fragment mésio-proximal de pointe de projectile. Dans le cadre du projet PaleoCet, l'identification taxinomique de l'artefact osseux a fait l'objet d'une empreinte peptidique de masse de collagène (PMF). Aussi connu sous le nom de ZooMS, le collagène PMF discrimine les groupes taxinomiques en fonction des différences dans la séquence protéique du collagène observées par spectrométrie de masse MALDI-TOF. Les sous-échantillons osseux de l'extérieur et de l'intérieur de l'objet ont produit des spectres MALDI-TOF similaires, que l'on peut rapporter à la baleine grise (*Eschrichtius robustus*). La pièce de La Madeleine est la première preuve de la présence de cette espèce dans les eaux atlantiques à la fin du MIS2, ce qui suggère une continuité entre les populations pléistocènes et holocènes de cette espèce. Elle constitue la plus ancienne trace de baleine grise identifiée en contexte archéologique, et est donc le plus ancien indice des interactions millénaires entre les humains et cette espèce de baleine très côtière. Une fabrication sur la côte basque, puis une circulation à travers les Pyrénées occidentales, le nord-est des Landes et la Gironde, jusqu'au Périgord (environ 350 km de trajet), pourrait être la voie la plus plausible suivie par la pièce de La Madeleine.

MOTS-CLÉS Industrie osseuse, analyse Zooms, Baleine grise (*Eschrichtius robustus*), La Madeleine, Magdalénien, pointe de projectile.

INTRODUCTION

Discovered in 1863 by E. Lartet and H. Christy, who carried out the first excavations there (1863-1865), the rockshelter of La Madeleine (Tursac, Dordogne, France; **fig. 1**) was the subject of various archaeological field projects (E. Massénat and P. Girod in 1895; E. Rivière in 1901) before D. Peyrony conducted methodical archaeological investigations there between 1910 and 1913 (Capitan and Peyrony 1928). The chronological (four radiocarbon dates) and environmental framework were subsequently described as field work was carried out between 1967 and 1983 under the direction of J.-M. Bouvier (Bouvier 1977, 1979).

The historical importance of La Madeleine goes well beyond the important archaeological assemblages recovered at the end of each excavation and now scattered in various museums around the world (Bouvier 1977: 80-82; Mémoire 1990). La Madeleine has been the eponymous site of the Magdalenian culture since 1889, and the bone industries from the Peyrony excavations served as a basis for H. Breuil (1912) to define the middle (IV) and upper (V and VI) stages of the Magdalenian.

During an integrated study of the bones and reindeer antlers kept at the Musée national de Préhistoire (Peyrony and Bouvier collections; Fontana 2017 and L. Fontana and F.-X. Chauvière, work in progress), the attention of one of us (F.-X. C.) was drawn to the object described in this article. The bone structure of this fragmentary projectile point, found classified among the shaped reindeer antlers, strongly resembled that of Cetacean bones seen in the Magdalenian series of Isturitz (Saint-Martin-d'Arberoue and Isturitz, Pyrénées-Atlantiques, France; collections from the Passemard and Saint-Périer excavations at the Musée d'archéologie nationale) and Duruthy (Sorde-l'Abbaye, Landes, France; collections from the Arambourou excavations at the Abbaye d'Arthous). This macroscopic identification would then be confirmed by J.-M. Pétillon and further clarified by physical-chemical analyses (Pétillon and Chauvière 2019 and see below).

Given the unprecedented nature of this taxonomic determination and the full development of current research on the exploitation of marine mammals in the Paleolithic period, it seemed essential to include an analysis of this object from La Madeleine. First, we will review studies on the exploitation of Cetaceans in the European Paleolithic. The artifact is then described in detail in all its technical and functional dimensions as well as from the point of view of its anatomical and above all specific provenance. We then report the results of biomolecular analyses to identify the species of Cetacean used to construct the artifact, and discuss the implications of the artifact within the framework of the recent Paleolithic.



— FIGURE 1 —

Sites and geographic names mentioned in the text. Triangles: sites that yielded objects made of whale bone; circles: other sites. 1: La Madeleine; 2: Laugerie-Basse; 3: Montgaudier; 4: Las Caldas; 5: Santa Catalina; 6: Isturitz; 7: Bourrouilla; 8: Duruthy; 9: Brassempouy; 10: Saint-Michel and Espalungue; 11: Espélugues; 12: Lortet; 13: Harpons; 14: Gourdan; 15: Tuc d'Audoubert; 16: Mas d'Azil; 17: La Vache. Not on map: Nerja, Andernach-Martinsberg. Map of Europe during the Last Glacial Maximum (maximum sea level recession at -120m, maximum extension of glaciers; map A. Sécher after data from Ehlers and Gibbard 2004; Farr et al. 2007; Becker et al. 2015; Zickel et al. 2016).

Sites et zones géographiques mentionnés dans le texte. Triangles : sites qui ont livré des objets en os de baleine ; cercles : autres sites. 1 : La Madeleine ; 2 : Laugerie-Basse ; 3 : Montgaudier ; 4 : Las Caldas ; 5 : Santa Catalina ; 6 : Isturitz ; 7 : Bourrouilla ; 8 : Duruthy ; 9 : Brassempouy ; 10 : Saint-Michel et Espalungue ; 11 : Espélugues ; 12 : Lortet ; 13 : Harpons ; 14 : Gourdan ; 15 : Tuc d'Audoubert ; 16 : Mas d'Azil ; 17 : La Vache. Hors carte : Nerja, Andernach-Martinsberg. Carte de l'Europe durant le dernier maximum glaciaire (recul maximal du niveau de la mer à -120 m, extension maximale des glaciers ; carte A. Sécher d'après des données d'Ehlers et Gibbard 2004 ; Farr et al. 2007 ; Becker et al. 2015 ; Zickel et al. 2016).

1 | THE EXPLOITATION OF CETACEANS IN THE EUROPEAN PALEOLITHIC

1.1 | History of research

For decades, the exploitation of seashore resources by paleolithic hunter-gatherers in Western Europe has received little academic attention. L. G. Straus and G. A. Clark were among the first, in the 1970s and the 1980s, to support the hypothesis that this exploitation became progressively more intense over the course of the Upper Paleolithic (Straus and Clark 1986). In later years, in France, the work of Y. Taborin stressed the importance of marine shells among Paleolithic personal ornaments (Taborin 1993), while two seminal papers addressed the issues of the exploitation of seals and the use of Cetacean teeth (Sonneville-Bordes and Laurent 1983; Poplin 1983), and the first review papers on the importance of the seashore in the Paleolithic began to appear (Cleyet-Merle and Madelaine 1995; Serangeli 2002, 2003). However, it is mostly from the 2000s on, in the wake of a seminal article (Erlandson 2001), that research on this topic intensified and led to an abundance of new data, most importantly

from sites in the Iberian Peninsula (among many others: Corchón et al. 2008; Álvarez-Fernández 2011; Haws et al. 2011; Fano et al. 2013; Berganza and Arribas 2014; etc.).

Despite this renewed interest, the currently available evidence demonstrates that the exploitation of marine resources in the Middle Paleolithic and the Early Upper Paleolithic (Aurignacian) was still sporadic, or occasional at best (Álvarez-Fernández 2015). A slight intensification is perceptible in the Middle Upper Paleolithic (Gravettian and Solutrean), at least in certain regions such as the Cantabrian coast where the exploitation of mollusks—mostly *Patella vulgata*—becomes more frequent (Álvarez-Fernández and Fernández-García 2011; Castaños and Álvarez-Fernández 2012). The bulk of the Paleolithic evidence, however, comes from the Late and Final Upper Paleolithic, corresponding to the Magdalenian and subsequent cultures. This evidence is particularly dense around the Gulf of Biscay (review in Pétilon 2016a) and in southernmost Iberia (e.g., Morales-Pérez et al. 2019), suggesting the existence of a true coastal economy. In these contexts, besides the exploitation of marine mollusks, fish and birds, the presence of sea mammals in the economic and symbolic worlds is documented through

remains and depictions of seals and Cetaceans (review in Pétillon 2018). Remains of Cetaceans are observed in the form of isolated teeth (sometimes carved and/or pierced), rarely bones (at Santa Catalina and Nerja), and fragments of whale barnacles (at Las Caldas and Nerja: these crustaceans are commonly found embedded in the skin of large Cetaceans and their presence indicates the transport of whale skin, blubber and perhaps meat from the coast to the site).

It is within this research context that a number of objects made of whale bone were identified in the bone industry of several Magdalenian sites. The use of this raw material was first acknowledged by F. Poplin and one of the present authors (J.-M. P.) at the site of Isturitz, in the Middle and Upper Magdalenian layers (Pétillon 2008). In the following years, several other specimens were identified in sites close to Isturitz: Saint-Michel d'Arudy (Pujol 2009), Brassempouy (Lefebvre 2014), and Duruthy (F.-X. C. and M. Dachary, pers. obs.). Subsequently, a systematic review of bone industry assemblages from the Magdalenian sites of the northern Pyrenees increased the number of objects to 109 and the number of sites to 12 (Isturitz, Duruthy, Brassempouy, Saint-Michel, Espalungue, Espéluques, Lortet, Harpons, Gourdan, Le Tuc d'Auboubert, Le Mas d'Azil, La Vache: Pétillon 2013). Very few objects of this type were found during recent excavations, and most of them are from ancient collections, for which few investigations had been made for Cetacean materials. The identification was based on macroscopic criteria (see below) but was also backed up by micro PIXE/PIGE analysis (Müller and Reiche 2011). Although they are sufficient for the generic identification of whale bone, these criteria do not allow us to identify either the species or the anatomical element exploited.

The large majority of the objects currently identified are related to weaponry: projectile points and foreshafts represent 74 of the 88 objects for which a typological attribution could be suggested, the others being wedges ($n = 9$), half-round rods ($n = 2$) or other types of finished objects ($n = 3$; Pétillon 2013). The choice of whale bone as a material for the manufacture of projectile heads can have several reasons. The most obvious is that these bones can be used to manufacture objects of very large dimensions (length, but also caliber, i.e. width/thickness). But it can also be noted that the impact resistance of whale bone, while not being as high as that of antler, is above that of most land mammal bones (see Margaris 2014 and references therein).

1.2 | Geographical and chronological distribution

The northern Pyrenean range is so far the only region in which the Magdalenian assemblages of osseous industry have been systematically investigated (with a few exceptions) in order to identify objects made of whale bone. Three quarters of these objects were shown to be concentrated in the Western Pyrenees, while the Central Pyrenees yielded the remaining 25% and none was found in the eastern part of the range (fig. 1). This contrasted distribution is not a function of the relative abundance of worked bone assemblages, and it led to the hypothesis that the Atlantic Coast—the current Basque seashore—was the most likely origin of the bone material exploited, even

if the putative seashore sites are of course no longer accessible today (Pétillon 2013). Outside the Pyrenean region, discoveries are much sparser: only the object from Andernach-Martinsberg (Langley and Street 2013), and one from Las Caldas (Corchón and Ortega 2017: 364), have been published.

Most objects made of whale bone are from Middle and Upper Magdalenian contexts. The associated ^{14}C dates range between ca. 17500 and 15000 cal BP, which corresponds to the late part of the Middle Magdalenian and the early part of the Upper Magdalenian; the typology of the objects is compatible with this attribution (Pétillon 2013). Objects made of whale bone attributed to earlier periods are very few, often unpublished, and their identification is sometimes uncertain. N. Goutas (2008: 49) mentions two objects made of whale bone from the Gravettian layers of Isturitz, but the micro PIXE/PIGE analysis of one of these objects did not confirm its identification as whale bone (Müller and Reiche 2011). Furthermore, no object made of whale bone has been directly radiocarbon-dated so far, and the dating of this material poses specific problems: the apparent age (“reservoir age”) of the Gulf of Biscay in the Magdalenian has not been precisely quantified; and the (theoretically possible) use of whale bones originating from individuals that had been stranded significantly earlier might add a further bias (for similar problems in other contexts see for example: Nelson and McGhee 2002; Friesen and Arnold 2008; Marchand *et al.* 2009).

1.3 | Recent developments: the PaleoCet project

The recurrent presence of evidence linked to the exploitation of whales suggests that the abundant resource represented by large Cetaceans might have been one of the major incentives for the intensification of the exploitation of the seashore in the Middle and Upper Magdalenian. Nevertheless, several limitations in our knowledge of whale exploitation prevent us from testing this hypothesis: none of the remains is identified to species level, which means that we have no idea of the range of whale taxa exploited by the Magdalenian foragers, and thus of their ecology and behavior, which can be markedly different from one species to another (Rodrigues *et al.* 2016; Bernal-Casasola *et al.* 2016); the chronology of the production and diffusion of whale products remains imprecise; and a systematic search for whale-bone objects outside the northern Pyrenees has not been undertaken, which means that we do not know if this production was a local phenomenon or part of a widely shared technical package.

The PaleoCet project was devised in order to overcome these limitations. Scheduled from 2019 to 2021, funded by the Agence nationale de la recherche and directed by J.-M. Pétillon and A. Zazzo, it is a joint project between the UMRs Traces (Toulouse) and AASPE (Paris), and includes collaborations with UMR Cefe (Montpellier), the University of British Columbia (Vancouver) and the University of York. It will include the taxonomic identification of a large number of Magdalenian whale remains through collagen peptide mass fingerprinting (see below); the direct radiocarbon dating of these remains with the ECHoMICADAS (Environnement, Climat et Homme, Mini Carbon Dating System, installed at Gif-sur-Yvette since

2017), which makes it possible to drastically reduce sample sizes (Synal 2013; Synal *et al.* 2007; Cersoy *et al.* 2017a, 2017b), and accompanied by a specific protocol to quantify the local reservoir effect; and the investigation of collections of bone industry outside the Pyrenees to assess the presence of worked bone remains. The object presented in this paper was identified and analyzed as a part of this project.

2 | THE BONE PROJECTILE POINT OF LA MADELEINE

2.1 | Stratigraphic and cultural context

The Cetacean bone artifact, the description of which will follow, is the result of excavations carried out under the direction of D. Peyrony. It bears the inscription «la mad. m. V». It was therefore discovered in the middle layer (“*couche moyenne*”) of the stratigraphy described by Capitan and Peyrony (1928: 63-69) and subsequently attributed to the “Magdalenian V” by H. Breuil (1912). This layer was interposed between the upper and lower layers of the rockshelter (“*couches inférieure et supérieure*”), which refer respectively to the “Magdalenian IV” and “Magdalenian VI” of Breuil’s classification (1912).

The middle layer corresponds to layers C25 to C20 of the stratigraphy established by J.-M. Bouvier (Fontana, 2017: 350). Two radiocarbon dates are currently available (C21: 12750 ± 240 BP, Ly 920; and C25: 13070 ± 190 BP, Ly 921; Bouvier 1979: 439).

Capitan and Peyrony do not mention this artifact in the chapter concerning the bone industry found in the middle layer (*op. cit.*: 65-69). They report the presence of barbed implements (harpoons with a single row of barbs, tridents), double bevelled projectile points, some conico-cylindrical based and some single-bevelled. They also note the presence of perforated batons, spearthrowers and half-round rods. The assemblage (1,227 pieces) was one of the subjects of a technical analysis of manufacturing techniques in L. Braëm’s Ph. D. Dissertation (Braëm 2008: 168-174 and following; Braëm-Villesuzanne 2016). In addition, there are engraved and sculpted pieces of portable art and ornaments (Capitan and Peyrony, *op. cit.*: 71-78 ; Taborin 1993 ; Vanhaeren and d’Errico 2001).

2.2 | Description and function

The artifact is in a very good state of preservation (**fig. 2**). It is a fully worked object, elongated in shape, with straight outline and profile, a circular cross-section and parallel sides. Its maximum dimensions are 129 X 13.2 X 12.8 mm and it weighs 18.6 g. One of its extremities, considered as distal (see below), is fractured. The proximal part is conical in shape, slightly flat, with a blunt end.

The initial identification of the raw material was based on the macroscopic observation of the characteristics of the bony surface. This surface shows indeed a porous aspect which is very different from that of the spongy tissue characteristic of land mammal bones or antler: its structure is intermediate between that of compact and cancellous bone, and could be described as compact bone

enclosing relatively wide and sparse trabeculae. The distal fracture of the artifact could have provided an opportunity to directly view these pores in section, but the fracture plane is incrustated with a sediment that makes this examination impossible. The porosity is rather evenly distributed along the whole length and thickness of the artifact: although the pores are denser on one of the sides (third view from the left on **fig. 2**), there is no clear-cut opposition between a “spongy” side and a “compact” one—which, here again, differs from the usual characteristics of both land mammal bone and antler. Such a structure is, however, typical of the bones of Cetaceans (Margaris 2006, 2014): these bones have no medullary cavity and, although surrounded by a cortical shell, are mostly composed of cancellous tissue (Felts and Spurrell 1965; Buffrénil and Schoevaert 1988; Campbell-Malone 2007). This structure is usually interpreted as a specific adaptation to buoyancy in deep water (Taylor 2000; Ricqlès and Buffrénil 2001; Gray *et al.* 2007). The small size of the object and its degree of transformation prevents us from identifying the anatomical element from which the bone blank was taken.

On such a porous material, traces of working are usually difficult to distinguish, and this object is no exception. The techniques of blank production cannot be identified: no trace of this early stage of manufacture is visible on the artifact. It can be assumed, however, that the shaping of the object was done by scraping, as the aspect of the surface is not compatible with what would have resulted from shaping by grinding. The object shows no surface features such as decoration, striations, longitudinal grooves, etc.

The extremity of the artifact displays a step-terminating beveled break. This morphology shows that the fracture is not post-depositional but was the result of forceful bending during use. This type of damage is compatible with use as a projectile point, but also with other functions involving strong longitudinal constraints (e.g., wedges: see discussion and references in Pétillon *et al.* 2016a). However, the extremity opposite the fracture shows no macroscopic usewear traces, and its shape is similar to that of points with massive base known elsewhere in the Magdalenian (see Pétillon 2016b and references therein). A typological identification as a mesial-proximal fragment of projectile point is thus the most likely hypothesis. According to a classic model (Chadelle *et al.* 1991), we can suppose that this point was broken in the field, that the mesio-proximal fragment was brought to the site still attached to a projectile shaft, and was then removed from its haft and discarded on site. The width and thickness of the artifact, ca. 13 mm, clearly hint at a projectile of large caliber, in the “spear” size range (as opposed to arrows: Cattelain 1997; Pétillon *et al.* 2011: 1273).

In the Magdalenian sites of the northern Pyrenean range, the points with massive base made of whale bone are found in contexts dating to the second half of the Middle Magdalenian rather than the beginning of the Upper Magdalenian (Pétillon 2008, 2013). This typo-chronological argument suggests that the artifact from La Madeleine could be slightly older than what its stratigraphic attribution indicates (in Breuil’s terms: “Magdalenian IV” rather than “Magdalenian V”?).

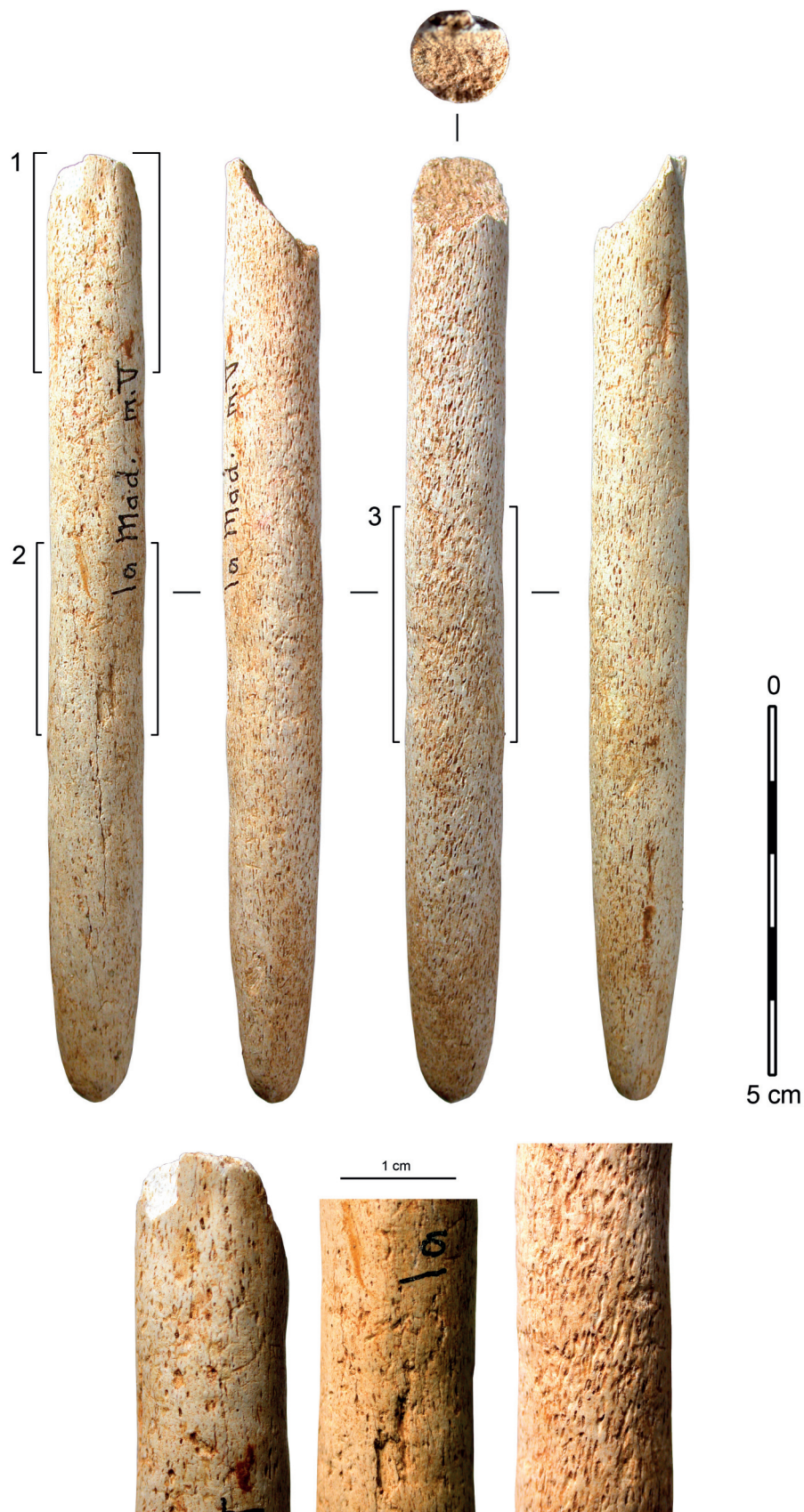


FIGURE 2

Whale-bone artifact from La Madeleine "M. V", Peyrony collection, Musée national de préhistoire, and details of the bony surface. Pictures J.-M. Pétillon.

Pointe de projectile en os de baleine grise (Eschrichtius robustus) de La Madeleine «M. V», collection Peyrony, Musée national de préhistoire ; détails de la surface osseuse. Clichés J.-M. Pétillon.

2.3 | Specific identification

Collagen peptide mass fingerprinting (PMF) was applied to taxonomically identify the bone artifact. Also known as ZooMS (zooarcheology by mass spectrometry), collagen PMF discriminates taxonomic groups based on differences in the collagen protein sequence observed through Matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF) mass spectrometry (Collins *et al.* 2010; Buckley *et al.* 2014).

In accordance with the routine procedure established in the PaleoCet project, a photogrammetric survey of the object was carried out before any sampling took place. The data necessary to create a 3D model of the artifact in its complete state will thus be archived, both in the project's database and at the museum, in order to keep a record of the artifact's morphology before sampling for conservation purpose. The object was photographed using a Nikon D5500 with a Nikon 60mm lens. This camera generates pictures of 24 megapixels (6k X 4k). The pictures are kept in raw format in order to avoid any degradation through filtering or compression. The object was photographed on a revolving plate in a light tent lit by two lamps. A large number of pictures were taken at different angles and at least twice: a "front side" series and a "back side" series. The object can thus be reconstructed in its entirety, without a "hidden side".

Sampling was done at the *Musée national de préhistoire* on March 28, 2019, using a Proxxon Colt 2 pocket drill equipped with a metal bit 2 mm in diameter. The distal fracture plane was selected as sampling spot and the artifact was drilled longitudinally to extract a powder sample while keeping the sampling as discreet as possible, especially since the object was intended for display in the exhibition "Animaux rares, gibiers inattendus. Reflets de

la biodiversité". ZooMS analysis typically requires ca. 10–30 mg of bone sampling. In this case, in order to detect and avoid any contamination that might be present on the surface of the artifact, the bone powder sampled from the surface of the artifact (approximately 10 mg) was separated from the one sampled from the interior (approximately 24 mg).

The two subsamples were prepared and analyzed at the BioArCh centre, University of York following the methods described in Evans *et al.* (2016). The two sub-samples of bone powder were demineralized using 0.6 M hydrochloric acid. The collagen was gelatinized in 100 µl of 50 mM ammonium bicarbonate at 65 °C for 1 h, and 50 µl of the supernatant was then incubated overnight with 0.4 µg of trypsin at 37 °C. The solution was then acidified to 0.1 % trifluoroacetic acid to deactivate the trypsin, and purified using a 100 µl C18 resin ZipTip pipette tip (EMD Millipore). Collagen from both samples was spotted in triplicate using a matrix of α -cyano-4-hydroxycinnamic acid along with calibration standards on a 384-spot MALDI target plate, and run on a Bruker ultraflex III MALDI TOF/TOF mass spectrometer at the University of York. The mMass software (Strohalm *et al.* 2008) was used to average spectra replicates from each subsample, which were then compared to the list of m/z markers for marine mammals presented in Kirby *et al.* (2013), Buckley *et al.* (2014) and Hufthammer *et al.* (2018), in order to taxonomically identify the species used as raw material for the artifact.

The bone subsamples from the exterior and interior of the artifact produced similar MALDI-TOF spectra, both of which could be confidently identified as gray whale (*Eschrichtius robustus*), based on gray whale specific markers presented in Kirby *et al.* (2013) and Hufthammer *et al.* (2018), specifically, the presence of diagnostic collagen peptide F at 2899 (fig. 3).

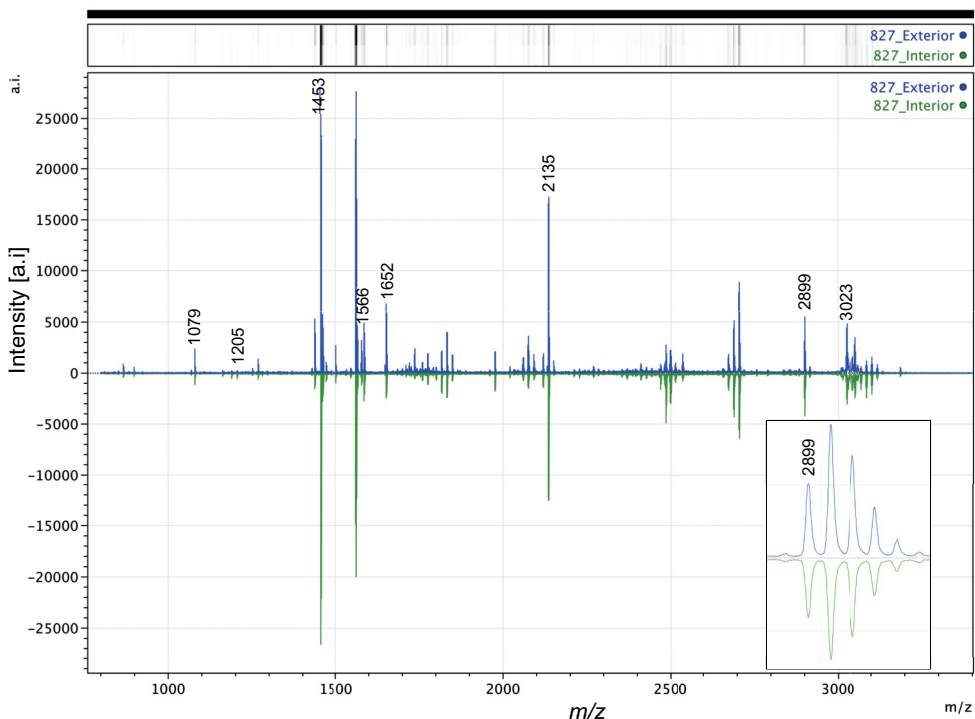


FIGURE 3

MALDI-ToF spectra for external (top) and internal (bottom) subsamples of the artifact, labelled with taxonomically informative peaks presented in Kirby *et al.* (2013), Buckley *et al.* (2014); inset displays close-up of the gray whale diagnostic Peptide F at m/z 2899. a.i. = absolute intensity; m/z = mass-to-charge ratio.

*Spectre MALDI-ToF des sous-échantillons externes (en haut) et internes (en bas) de l'objet, marqués par des pics taxonomiques caractéristiques présentés dans Kirby *et al.* (2013), Buckley *et al.* (2014); l'encadré montre un gros plan du Peptide F diagnostique pour la baleine grise au m/z 2899. a.i. = intensité absolue; m/z = ratio masse/charge.*

3 | DISCUSSION

This study further highlights utility of ZooMS to taxonomically identify worked bone artifact assemblages, dating back to the Upper Paleolithic. Previous research successfully applied ZooMS to reveal raw material selection and manufacture strategies of Medieval bone and antler combs from Scandinavia (Von Holstein *et al.* 2014; Brandt *et al.* 2018), Iroquois bone points from Eastern North America (McGrath *et al.* 2019), and bone tools from the North African Later Stone Age cave site of Taforalt (Desmond *et al.* 2018). These combined studies highlight the myriad advantages of ZooMS when working with highly modified, morphologically indistinguishable artifacts. Unlike more conventional DNA ‘barcoding approaches’, which require some prior knowledge of species in order to design appropriate primers, ZooMS can be applied to highly fragmented non-diagnostic bone without any prior taxonomic knowledge. Moreover, ZooMS targets collagen, which has the potential to survive at least 10 times longer than DNA, increasing the likelihood of analytical success (Welker *et al.* 2015), especially for minute samples such as those frequently taken from culturally significant items. ZooMS requires extremely small sample sizes (frequently >5-10 mg of bone) and recent studies have indicated that sufficient collagen may be recovered using a non-destructive ammonium bicarbonate buffer (van Doorn *et al.* 2011), by gentle rubbing with an eraser (Fiddymont *et al.* 2015) or through the analysis of artifact storage bags (McGrath *et al.* 2019) which can enable artifacts of high cultural value to be sampled non-invasively.

Strictly speaking, the geographic origin of this bone cannot be ascertained, but certain assumptions can be made. A Mediterranean origin cannot be ruled out, and would imply travel distances ca. 400 km given the recession of the coast in the Gulf of Lion at that time. However, the distribution of the other whale-bone objects known today from Magdalenian sites points to an Atlantic origin (Pétillon 2013); and this context is an argument in favor of a similar origin for the artifact from La Madeleine. In this perspective, in the Upper Paleolithic the shortest route from the Atlantic seashore to La Madeleine shelter would have been from the coast of present-day Gironde and Landes, at orthodromic distances ca. 240 km given the recession of the sea level (fig. 1). However, the likely date of the La Madeleine object is either the Recent Middle Magdalenian or the Early Upper Magdalenian, ca. 17.5-15 cal ka BP (a period which is also that of the diffusion of whale-bone objects in the Pyrenees: Pétillon 2013); and recent studies support the hypothesis that, at that period, the Gironde was devoid of Magdalenian sites, and this region was probably largely deserted because of the harsh conditions of the Heinrich Stadial 1a (Barshay-Szmidt *et al.* 2016; Naughton *et al.* 2016; Pétillon *et al.* 2016b). The same holds true for the Landes sandy region, which has been repulsive for human groups during a large part of the Paleolithic because of its desert conditions (Bertran *et al.* 2013; Sitzia 2014; Sitzia *et al.* 2015); subsequent research suggests that a similar situation prevailed further east in the Garonne valley (Bruxelles and Jarry 2011). Thus, the shortest route between La Madeleine and the Atlantic coast crosses regions that were very probably barren and inhospitable at these times, and for this reason this route might actually not be the most likely to have been taken

by the whale-bone diffusion networks. Conversely, in the western and central part of the northern Pyrenean range are numerous sites dated to that period, a dozen of which yielded objects made of whale bone; and many studies pinpointed evidence of communication between this region and the Magdalenian Périgord (circulation of lithic raw materials, distribution of specific technical and graphic traits, etc.: e.g., Cattelain 2005; Fritz *et al.* 2007). This is especially true for the Magdalenian assemblages from La Madeleine and Laugerie-Basse shelters, which share many common points with contemporaneous sites in the Pyrenees (e.g., collective 2014; Cattelain and Bellier 2014; Cattelain 2016). The manufacturing of the La Madeleine artifact on the Basque coast, followed by its circulation through the western Pyrenees and then towards the north at the east of Landes and Gironde up to Périgord (a travel distance of roughly 350 km), might thus actually be the most plausible route.

Before the determination of this whale-bone artifact in La Madeleine, only shells were used to link this archaeological site – directly or indirectly – with the Paleolithic coastlines. In the “middle layer” (Magdalenian V) of the stratigraphy described by Capitan and Peyrony, Y. Taborin mentions the presence of Würmian shells, either Atlantic or Mediterranean: several *Turritella communis*, 1 or 2 *Cardium edule* (perforated), 1 *Pecten maximus* or *jacobus* (Taborin 1993, pp. 158-159, 432, 485; see also Vanhaeren and Errico, op. cit., p. 206-207).

Irrespective of whether gray whales were actively captured or scavenged from stranded individuals, their coastal ecology means that they must have been well known to coastal communities, which is important to consider when envisioning their place in the Magdalenian cultural world. Contrary to high sea species, which were probably only known from dead or moribund individuals, live gray whales would have been observed from high points in the coastline or as the whales jumped. Gray whales are medium-sized baleen whales (mysticetes) measuring 13-15 m with a weight ca. 14-35 tons. They are named after the gray-white patches on their dark skin. Anatomically this species – the only living one in the genus *Eschrichtiidae* – is characterized by the absence of dorsal fin; short, whitish baleen plates; and a double blowhole that can produce distinctive heart-shaped blows (Swartz 2014). They are migratory, moving seasonally from cold high-latitude feeding grounds to warmer lower-latitude calving areas. They are coastal in all of these seasons, feeding in low-depth waters, migrating by hugging the coastline, and calving in sheltered low-depth lagoons. Whereas in today’s climate the Gulf of Biscay and the Mediterranean would more likely correspond to calving grounds (Rodrigues *et al.* 2018), it is possible that in the Upper Paleolithic they would have corresponded to feeding grounds. In any case, they would have been conspicuously present at predictable seasons, and they are likely to have been part of the range of animals whose ecology would have been known to Paleolithic hunter-gatherers.

Gray whales today have a strictly North Pacific distribution: they do not seem to have survived in the North Atlantic after the eighteenth century, having probably disappeared due to whaling even if the circumstances of their disappearance remain poorly understood (Rodrigues *et al.* 2018). Previous evidence of their past presence in the

North Atlantic dates either to the Holocene or to the middle of MIS3, before 35 cal ka BP, with a gap in-between (Alter *et al.* 2015; Garrison *et al.* 2019). Furthermore, molecular analyses revealed little genetic continuity between the late Pleistocene and the Holocene populations: most Atlantic Holocene specimens analyzed by Alter *et al.* (2015) are genetically closer to Holocene Pacific gray whales than to Pleistocene Atlantic specimens, leading these authors to suggest that the majority of the Atlantic Holocene population were the result of a second colonization event from the Pacific, after warming temperatures, sea-level rise, and decreases in sea ice permitted passage through the Bering Strait (Alter *et al.*, 2015). The artifact from La Madeleine is the first evidence of the presence of this species in Atlantic waters at the end of MIS2, thus supporting instead a continuity between the Pleistocene and the Holocene populations.

4 | CONCLUSION

Prior to this study, the identification of whale species in the Magdalenian was based almost exclusively on the few teeth remains found in Magdalenian sites and on tentative taxonomic attributions of the rare whale depictions in the Magdalenian cave art and portable art (see review and references in Pétillon 2018). These species include Sperm Whale (teeth from Mas d’Azil and Las Caldas, possible depictions in Bourrouilla, La Vache and Las Caldas), Long-Finned Pilot whale (teeth in Las Caldas and possibly in Duruthy, Arambourou collection) and, without any certainty, Beluga whale (possible teeth in Duruthy, Arambourou collection; possible depiction in Las Caldas?) and one species of Right whale (presence of specific whale barnacles at Nerja: Álvarez Fernández *et al.* 2014). The presence of Gray Whale had been suggested only once, based on the possible identification of small figures on the Montgaudier *bâton percé* as right whales or gray whales (Robineau 1984; one might add that the same suggestion can be made for the “fish” depicted on the same object). The analysis of the La Madeleine object is thus the first formal identification of this species in the Magdalenian, and in the European Paleolithic altogether. It shows the great potential of the Magdalenian assemblage of whale-bone object as a depository of whale biodiversity at that period, and the relevance of the ZoomS technique to bring that potential to realization.

Finally, this gray whale bone artifact represents (to our knowledge) the oldest identified gray whale in an archaeological context, being thus the oldest evidence of the millennia-long relationship between our two species.

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