Supplementary Information for

"Late Paleolithic whale bone tools reveal human and whale ecology in the Bay of Biscay"

Krista McGrath^{1α}, Laura G. van der Sluis^{2,3α}, Alexandre Lefebvre^{4,5}, Anne Charpentier⁶, Ana Rodrigues⁶, Esteban Álvarez-Fernández⁷, François Baleux⁸, Eduardo Berganza⁹, François-Xavier Chauvière¹⁰, Morgane Dachary^{8,11}, Elsa Duarte Matías¹², Claire Houmard¹³, Ana B. Marín-Arroyo⁴, Marco de la Rasilla Vives¹², Jesus Tapia⁹, François Thil¹⁴, Olivier Tombret², Leire Torres-Iglesias⁴, Camilla Speller¹⁵, Antoine Zazzo² & Jean-Marc Pétillon⁸*

¹Department of Prehistory and Institute of Environmental Science and Technology (ICTA), Universidad Autònoma de Barcelona, Bellaterra, Spain. ²BioArchéologie, Interactions Sociétés Environnements (BioArch), UMR 7209, Muséum national d'Histoire naturelle, CNRS, Paris, France. ³Department of Evolutionary Anthropology, University of Vienna, Vienna, Austria. Human Evolution and Archaeological Sciences (HEAS), Vienna, Austria. ⁴Grupo I+D+i EVOADAPTA, Universidad de Cantabria, Santander, Spain. ⁵De la Préhistoire à l'Actuel : Culture, Environnement et Anthropologie (PACEA), UMR 5199, CNRS, Université de Bordeaux, Pessac, France. 6CEFE, Univ Montpellier, CNRS, EPHE, IRD, Montpellier, France. ⁷GIR PREHUSAL, Universidad de Salamanca, Facultad de Geografía e Historia, Departamento de Prehistoria, Historia Antigua y Arqueología, Salamanca, Spain. 8Travaux et Recherches Archéologiques sur les Cultures, les Espaces et les Sociétés (TRACES) UMR 5608, CNRS, Université Toulouse Jean Jaurès, Toulouse, France. 9Sociedad de Ciencias Aranzadi, Donostia-San Sebastian, Spain. 10Office du patrimoine et de l'archéologie du canton de Neuchâtel, section Archéologie, Laténium, Hauterive, Switzerland. ¹¹Ministère de la Culture, Service Régional de l'Archéologie de Nouvelle-Aguitaine, Limoges, France. ¹²Departamento de Historia, Universidad de Oviedo, Oviedo, Spain. ¹³Université de Franche-Comté, UMR 6249 Chrono-environnement, Besançon, France. 14Laboratoire des Sciences du Climat et de l'Environnement (LSCE/IPSL), UMR 8212, CEA-CNRS-UVSQ, Université Paris Saclay, Gif-sur-Yvette, France. 15 Department of Anthropology, University of British Columbia, Vancouver, Canada.

This pdf file includes:

- Supplementary Figures 1 through 102.
- Supplementary Discussion: Comparing stable isotope signatures in Paleolithic versus modern whales
- Supplementary References: references cited in the captions of Supplementary Figures 99 through 102, or in the Supplementary Discussion.

^a These authors contributed equally.

^{*} Corresponding author: jean-marc.petillon@cnrs.fr

Supplementary Figures 1 through 90: Catalogue.

This catalogue includes the pictures of:

- All the worked objects whose identification as whale bone was confirmed by ZooMS, except: study numbers 590, 625 and 629, whose pictures could not be taken at the museum; study numbers 993, 994 and AN2 25/83-73 N17, published in other articles (Langley and Street 2013; Lucas et al. 2023).
- A sample of 26 unworked whale bone fragments from Santa Catalina, with a priority given to those that have an individual excavation number (as opposed to those found during screening, identified only through their spit number), and that were both analyzed with ZooMS and radiocarbon-dated.

Unless otherwise stated, the scale is 1:1, and the pictures were taken before sampling.

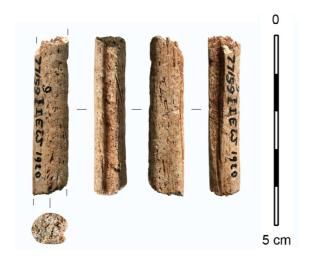
All pictures by A. Lefebvre and J.-M. Pétillon.

Supplementary Figure 1

Study number: 2

Museum number: MAN 7715919, I E omega 1920 Site and layer: Isturitz, salle de Saint-Martin, SI/E omega

Typology: projectile point ZooMS ID: Cetacean + cow glue contamination ¹⁴C date: none



Supplementary Figure 2

Study number: 4

Museum number: 4
Museum number: MAN 77159I6, I E St alpha 1914
Site and layer: Isturitz, Grande Salle, II/E
Typology: projectile point
ZooMS ID: Sperm whale

14C date: MUSE21043, 14260 ± 60 BP, 17268-16703 cal BP



Supplementary Figure 3 Study number: 6

Museum number: MAN Ist I (S) 1928, C2RMF: A
Site and layer: Isturitz, salle de Saint-Martin, SI/E omega
Typology: projectile point with massive base
ZooMS ID: Right whale / Bowhead whale

14C date: MUSE21044, 14290 ± 60 BP, 17299-16746 cal BP



Supplementary Figure 4
Study number: 7
Museum number: MAN Ist SI 1929, C2RMF: K
Site and layer: Isturitz, salle de Saint-Martin, SI/E omega
Typology: wedge
ZooMS ID: Cetacean + cow glue contamination

14C date: none



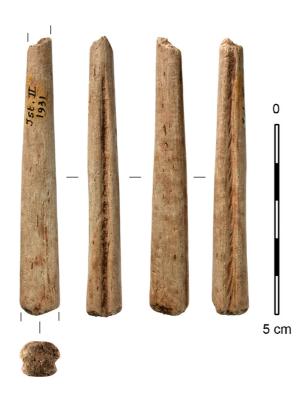
Supplementary Figure 5

Study number: 10

Museum number: MAN Ist II 1931 Site and layer: Isturitz, Grande Salle, II/E

Typology: projectile point
ZooMS ID: Sperm whale + cow glue contamination

14C date: none

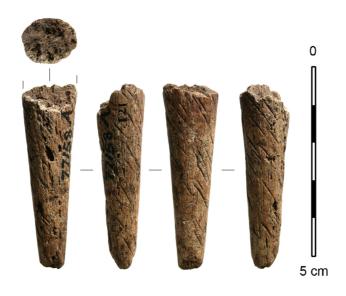


Supplementary Figure 6

Study number: 13

Museum number: MAN 77158A121 Site and layer: Isturitz, Grande Salle, II/E? Typology: projectile point with massive base ZooMS ID: Sperm whale

14C date: MUSE21045, 15110 ± 70 BP, 18248-17742 cal BP



Supplementary Figure 7
Study number: 14
Museum number: MAN Ist II 1932
Site and layer: Isturitz, Grande Salle, II/E
Typology: projectile point with massive base
ZooMS ID: Sperm whale + cow glue contamination
14C date: none



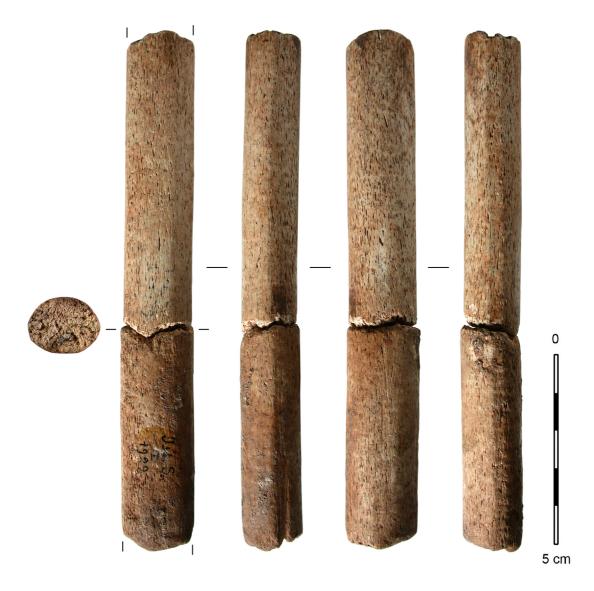
Supplementary Figure 8
Study number: 15
Museum number: MAN 77153A5, IE alpha 1914
Site and layer: Isturitz, Grande Salle, II/E
Typology: projectile point with massive base
ZooMS ID: Blue whale

14C date: MUSE21046, 15530 ± 80 BP, 18718-18198 cal BP



Supplementary Figure 9
Study number: 19
Museum number: MAN Ist SI 1929, C2RMF: J
Site and layer: Isturitz, salle de Saint-Martin, SI/E omega
Typology: object on splinter
ZooMS ID: Cetacean + cow glue contamination

14C date: none



Supplementary Figure 10 Study number: 36

Study number: 36
Museum number: MAN 77158B132
Site and layer: Isturitz, Grande Salle, II/E?
Typology: projectile point
ZooMS ID: Right whale / Bowhead whale

14C date: MUSE21047, 14250 ± 60 BP, 17256-16688 cal BP



Supplementary Figure 11

Study number: 38

Museum number: MAN Ist II 1934 Site and layer: Isturitz, Grande Salle, II/E Typology: projectile point with massive base ZooMS ID: Sperm whale + cow glue contamination ¹⁴C date: none



Supplementary Figure 12 Study number: 39

Museum number: MAN Ist II 1937 Site and layer: Isturitz, Grande Salle, II/E Typology: projectile point with massive base ZooMS ID: Fin whale + cow glue contamination

¹⁴C date: none



Supplementary Figure 13

Study number: 58

Museum number: Ist GD 2007 déblais paroi est, A Site and layer: Isturitz, salle de Saint-Martin, GD C2

Typology: projectile point ZooMS ID: Gray whale ¹⁴C date: MUSE21048, failed



Supplementary Figure 14

Study number: 59

Museum number: Ist GD 2008 B4, B

Site and layer: Isturitz, salle de Saint-Martin, GD C2

Typology: projectile point ZooMS ID: Fin whale

¹⁴C date: MUSE21049, failed



Supplementary Figure 15

Study number: 60

Museum number: Ist GD 2009 B3

Site and layer: Isturitz, salle de Saint-Martin, GD C2

Typology: projectile point

ZooMS ID: Cetacean (Sperm whale?)

¹⁴C date: MUSE21050, failed



Supplementary Figure 16 Study number: 82

Museum number: Ist GD 2010 B5

Site and layer: Isturitz, salle de Saint-Martin, GD C2
Typology: projectile point
ZooMS ID: Sperm whale

14C date: MUSE21051, failed



Supplementary Figure 17 Study number: 589

Museum number: MAN 77163G113 + 77162E20, IF1 (on fgt 77162E20) Site and layer: Isturitz, Grande Salle, I/F1 Typology: foreshaft ZooMS ID: Fin whale

14C date: MUSE 21058, 13820 ± 60 BP, 16729-16144 cal BP



Supplementary Figure 18 Study number: 630

Museum number: MAN Ist II 1931 + Ist II 1931 Site and layer: Isturitz, Grande Salle, II/E

Typology: foreshaft
ZooMS ID: Sperm whale + cow glue contamination

14C date: none

(scale 1:2)



Supplementary Figure 19 Study number: 634

Museum number: MAN 77158B362 + 77158B18
Site and layer: Isturitz, Grande Salle, II/E?
Typology: foreshaft
ZooMS ID: Sperm whale + cow glue contamination

14C date: none



Supplementary Figure 20
Study number: 637
Museum number: MAN 77159D10, IE 1914
Site and layer: Isturitz, Grande Salle, II/E
Typology: foreshaft
ZooMS ID: Sperm whale + cow glue contamination

14C date: none



Supplementary Figure 21
Study number: 749
Museum number: MAN Ist II 1930 + Ist I 1930
Site and layer: Isturitz, Grande Salle, I/F1 + II/E
Typology: object on splinter
ZooMS ID: Fin whale + cow glue contamination
14C date: none



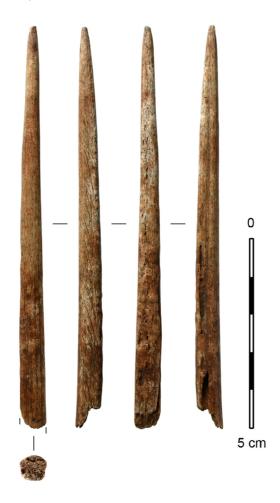
Supplementary Figure 22
Study number: 780
Museum number: MAN Ist I 1930
Site and layer: Isturitz, Grande Salle, I/F1
Typology: wedge
ZooMS ID: Sperm whale + cow glue contamination

14C date: none



Supplementary Figure 23
Study number: 781
Museum number: MHNT
Site and layer: Isturitz
Typology: projectile point
ZooMS ID: Sperm whale

14C date: MUSE 21054, 14430 ± 60 BP, 17446-16911 cal BP



Supplementary Figure 24

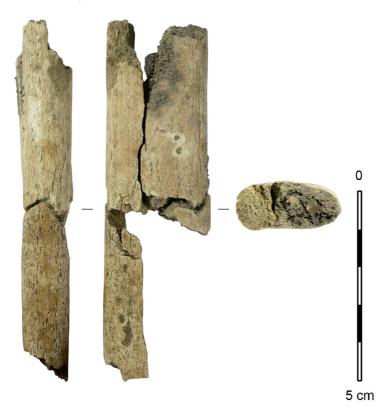
Study number: 783

Museum number: MAN 83642 + 83643, F28/29 + K69

Site and layer: Vache 3 & 4

Typology: unidentified
ZooMS ID: Right whale / Bowhead whale

14C date: MUSE20137, 14100 ± 60 BP, 17046-16501 cal BP



Supplementary Figure 25 Study number: 785

Museum number: MAN 83643, F8

Site and layer: Vache 4

Typology: wedge
ZooMS ID: Right whale / Bowhead whale

14C date: MUSE 20138, 14020 ± 60 BP, 16967-16402 cal BP



Supplementary Figure 26 Study number: 798

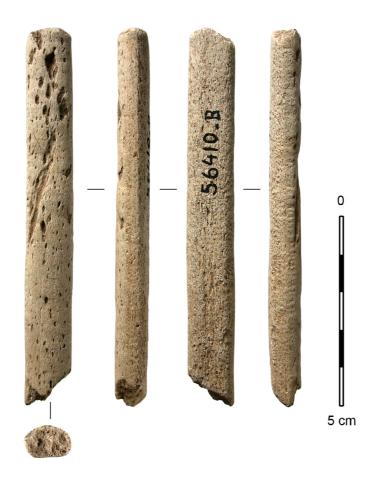
Museum number: 796
Museum number: MAN 56719, C2RMF: H1
Site and layer: Saint-Michel
Typology: projectile point with massive base
ZooMS ID: Right whale / Bowhead whale

14C date: MUSE20139, 14620 ± 60 BP, 17744-17114 cal BP



Supplementary Figure 27
Study number: 799
Museum number: MAN 56410, B
Site and layer: Saint-Michel
Typology: unidentified
ZooMS ID: Sperm whale

14C date: MUSE21042, 14450 ± 60 BP, 17477-16930 cal BP



Supplementary Figure 28
Study number: 815
Museum number: MH 55.33.9.1
Site and layer: Tuc d'Audoubert
Typology: foreshaft
ZooMS ID: Sperm whale

14C date: none
(scale 1:2)



Supplementary Figure 29
Study number: 816
Museum number: MHNT 17
Site and layer: Mas d'Azil
Typology: blank?
ZooMS ID: Sperm whale

14C date: MUSE20131, 14440 ± 90 BP, 17532-16875 cal BP



Supplementary Figure 30
Study number: 817
Museum number: MHNT MAZ 99.23.24
Site and layer: Mas d'Azil
Typology: projectile point with massive base
ZooMS ID: Sperm whale

14C date: MUSE21038, 14040 ± 60 BP, 16987-16428 cal BP



Supplementary Figure 31
Study number: Hum5
Museum number: 3945
Site and layer: Mas d'Azil
Typology: unidentified
ZooMS ID: Sperm whale

14C date: R00215.1, 14240 ± 190 BP, 16388-17462 cal BP



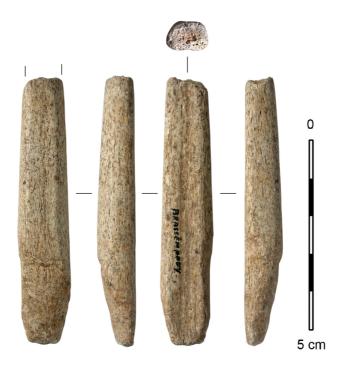
Supplementary Figure 32

Study number: 820

Museum number: MDW Brassempouy, Dubalen collection

Site and layer: Brassempouy, grotte du Pape Typology: shaping/maintenance waste?

ZooMS ID: Gray whale ¹⁴C date: MUSE21039, 13940 ± 60 BP, 16884-16299 cal BP



Supplementary Figure 33

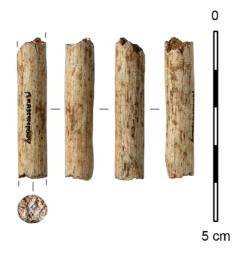
Study number: 821

Museum number: MDW Brassempouy, Dubalen collection

Site and layer: Brassempouy, grotte du Pape

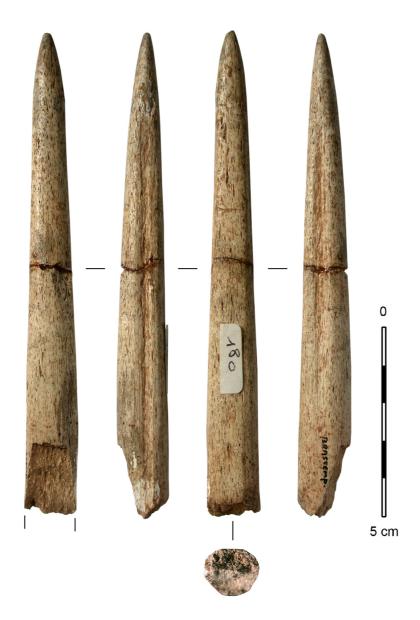
Typology: unidentified ZooMS ID: Sperm whale

¹⁴C date: MUSE21040, 13660 ± 60 BP, 16514-15946 cal BP



Supplementary Figure 34
Study number: 822
Museum number: MDW Brassempouy, Dubalen collection
Site and layer: Brassempouy, grotte du Pape
Typology: projectile point
ZooMS ID: Fin whale

14C date: MUSE21041, 14150 ± 70 BP, 17119-16536 cal BP



Supplementary Figure 35
Study number: 827
Museum number: MNP
Site and layer: Madeleine, Magd. V
Typology: projectile point with massive base
ZooMS ID: Gray whale

14C date: none



Supplementary Figure 36
Study number: 828
Museum number: CDP 79.1.267, S CV couche 2 (réattr. 3) 224
Site and layer: Duruthy 4
Typology: projectile point
ZooMS ID: Gray whale

14C date: MUSE20134, 14870 ± 90 BP, 18062-17379 cal BP
(scale 1:2)



Supplementary Figure 37
Study number: 829
Museum number: CDP 79.1.77, S BV couche 1
Site and layer: Duruthy 3
Typology: unidentified
ZooMS ID: Fin whale

14C date: MUSE20135, 14470 ± 90 BP, 17584-16913 cal BP (photograph taken after sampling)



Supplementary Figure 38
Study number: 830
Museum number: CDP 79.1.18469, S BVI couche 1 n°48
Site and layer: Duruthy 3
Typology: unidentified
ZooMS ID: Right whale / Bowhead whale

14C date: MUSE20136, failed
(photograph taken after sampling)



Supplementary Figure 39
Study number: 988
Museum number: CDP box 288-1
Site and layer: Duruthy 4
Typology: projectile point
ZooMS ID: Gray whale

14C date: MUSE21107, 14330 ± 70 BP, 17346-16784 cal BP



Supplementary Figure 40 Study number: 968

Museum number: ER III 13 (100-120)
Site and layer: Ermittia III
Typology: projectile point with massive base
ZooMS ID: Gray whale with polymer peaks

14C date: MUSE21099, 14420 ± 50 BP, 16915-17416 cal BP



Supplementary Figure 41
Study number: 969
Museum number: ER III 85
Site and layer: Ermittia III
Typology: projectile point
ZooMS ID: Gray whale

14C date: MUSE21100, 14520 ± 60 BP, 17004-17604 cal BP



Supplementary Figure 42
Study number: 970
Museum number: UR 9D 350
Site and layer: Urtiaga D
Typology: unidentified
ZooMS ID: Gray whale

14C date: MUSE21101, 14080 ± 50 BP, 16492-17017 cal BP



Supplementary Figure 43
Study number: 971
Museum number: ER III 4 (75-97)
Site and layer: Ermittia III
Typology: projectile point
ZooMS ID: Right whale / Bowhead whale

14C date: MUSE21102, 14470 ± 60 BP, 16950-17513 cal BP (scale 1:2)



Supplementary Figure 44
Study number: 973
Museum number: JU 890014#3
Site and layer: El Juyo 8
Typology: blank?
ZooMS ID: Fin whale

14C date: MUSE21104, 16290 ± 70 BP, 18992-19563 cal BP



Supplementary Figure 45
Study number: 974
Museum number: Ra CE 11473
Site and layer: Rascaño IVb
Typology: object on splinter
ZooMS ID: Fin whale

14C date: MUSE21105, 16770 ± 70 BP, 19569-20187 cal BP



Supplementary Figure 46 Study number: 975

Museum number: 8638
Site and layer: Las Caldas, Sala II, IIB
Typology: object on splinter
ZooMS ID: Sperm whale

14C date: MUSE21106, 13700 ± 50 BP, 16010-16549 cal BP



Supplementary Figure 47 Study number: 976 Museum number: 8640

Site and layer: Las Caldas, Sala II, IIIc-IV Typology: projectile point with double bevel ZooMS ID: Sperm whale ¹⁴C date: none



Supplementary Figure 48 Study number: 977

Museum number: 8644

Site and layer: Las Caldas, Sala II, V

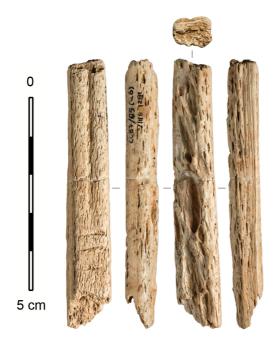
Typology: object on splinter ZooMS ID: Sperm whale

¹⁴C date: none



Supplementary Figure 49
Study number: 978
Museum number: 8645
Site and layer: Las Caldas, Sala II, VIIIb-IX
Typology: projectile point
ZooMS ID: Cetacean (poor spectra)

14C date: none



Supplementary Figure 50 Study number: 979 Museum number: 8646

Site and layer: Las Caldas, Sala II, VIII

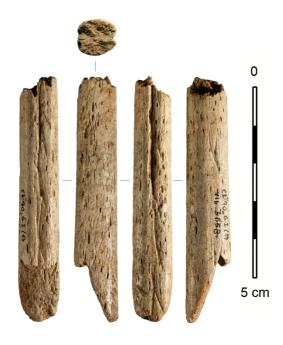
Typology: projectile point ZooMS ID: Dall's/Harbour porpoise (Phocoenidae) ¹⁴C date: none



Supplementary Figure 51 Study number: 980 Museum number: 8647

Site and layer: Las Caldas, Sala II, VIb
Typology: projectile point
ZooMS ID: Blue whale

14C date: none



Supplementary Figure 52 Study number: 981 Museum number: 8648

Site and layer: Las Caldas, Sala II, VIII

Typology: projectile point ZooMS ID: Sperm whale ¹⁴C date: none



Supplementary Figure 53 Study number: 982

Museum number: 8650

Site and layer: Las Caldas, Sala II, VI

Typology: foreshaft?
ZooMS ID: Sperm whale

14C date: none

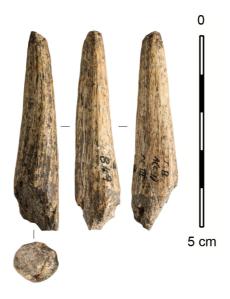


Supplementary Figure 54

Study number: 983 Museum number: 8658

Site and layer: Tito Bustillo, Área de Estancia, III

Typology: projectile point ZooMS ID: Sperm whale ¹⁴C date: none



Supplementary Figure 55
Study number: 984
Museum number: 8666
Site and layer: Las Caldas, Sala II, VIII
Typology: unidentified
ZooMS ID: Right whale/Bowhead whale

14C date: none

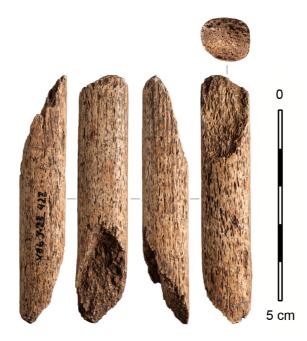


Supplementary Figure 56 Study number: 985 Museum number: 8699 Site and layer: La Viña IV Typology: projectile point ZooMS ID: Gray whale ¹⁴C date: none



Supplementary Figure 57 Study number: 986 Museum number: 8701 Site and layer: La Viña IV
Typology: object on splinter
ZooMS ID: Gray whale

14C date: none



Supplementary Figure 58
Study number: 987
Museum number: 8703
Site and layer: La Viña IV
Typology: projectile point?
ZooMS ID: Sperm whale

14C date: none



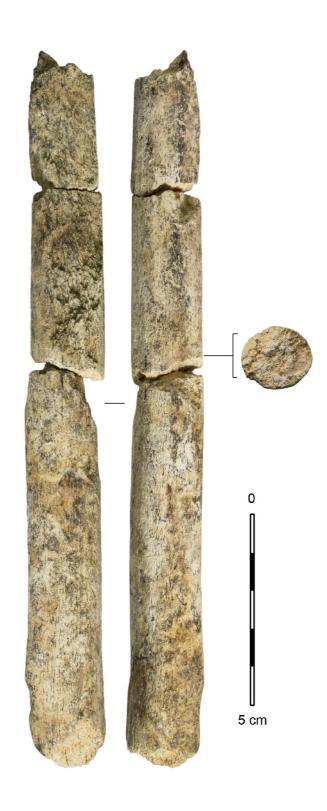
Supplementary Figure 59
Study number: Hum1
Museum number: TB-74
Site and layer: Tito Bustillo, Área de Estancia, 1B
Typology: blank?
ZooMS ID: Sperm whale

14C date: none



Supplementary Figure 60
Study number: Hum4
Museum number: 14-VII-98
Site and layer: Iruroin
Typology: object on splinter
ZooMS ID: Sperm whale

14C date: UCIAMS289875, 13880 ± 50 BP, 16219-16780 cal BP



Supplementary Figure 61

Study number: Hum8 Museum number: 6889 Site and layer: La Paloma 6 Typology: projectile point ZooMS ID: Sperm whale

14C date: UCIAMS289876, 14420 ± 60 BP, 16887-17415 cal BP



Supplementary Figure 62 Study number: Hum9 Museum number: 6761 Site and layer: La Paloma 4 Typology: projectile point ZooMS ID: Fin whale ¹⁴C date: R00217.1, 14284 ± 46 BP, 16730-17265 cal BP



Supplementary Figure 63

Study number: Hum14

Museum number: MNP-2012-3-1-12

Site and layer: Bourrouilla, déblais fouilles clandestines

Typology: unidentified ZooMS ID: Fin whale

14C date: R00222.1, 14046 ± 110 BP, 16340-17056 cal BP



Supplementary Figure 64

Study number: Hum18

Museum number: PBII-75/16/2/76

Site and layer: El Pendo II Typology: projectile point ZooMS ID: Sperm whale ¹⁴C date: R00225.1, 15549 ± 56 BP, 18222-18703 cal BP



Supplementary Figure 65 Study number: SC A6 127 68 Museum number: SC A6 127 68 Site and layer: Santa Catalina III

Typology: unworked
ZooMS ID: Right whale / Bowhead whale

14C dates:

MUSE21034, 13800 ± 60 BP, 16701-16119 cal BP MUSE20050, 13770 ± 60 BP, 16657-16081 cal BP MUSE20051, 13670 ± 60 BP, 16527-15959 cal BP

(scale 1:4)



Supplementary Figure 66 Study number: SC A6 130 15 Museum number: SC A6 130 15 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale

¹⁴C date: MUSE20065, 12790 ± 70 BP, 15385-14803 cal BP

(photograph taken after sampling)



Supplementary Figure 67 Study number: SC A6 L31G 2971 Museum number: SC A6 L31G 2971 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Right whale / Bowhead whale

¹⁴C date: MUSE20049, 13720 ± 90 BP, 16643-15972 cal BP



Supplementary Figure 68
Study number: SC A8 118 63
Museum number: SC A8 118 63
Site and layer: Santa Catalina III
Typology: unworked
ZooMS ID: Fin whale

14C date: MUSE20037, 13040 ± 50 BP, 15672-15153 cal BP



Supplementary Figure 69 Study number: SC A8 83 486 Museum number: SC A8 83 486 Site and layer: Santa Catalina II

Typology: unworked ZooMS ID: Fin whale ¹⁴C date: MUSE20034, 12700 ± 70 BP, 15259-14609 cal BP



Supplementary Figure 70 Study number: SC B4 87 76 Museum number: SC B4 87 76 Site and layer: Santa Catalina II

Typology: unworked ZooMS ID: Fin whale

¹⁴C date: MUSE20035, 12990 ± 50 BP, 15611-15096 cal BP



Supplementary Figure 71 Study number: SC B4 87 77 Museum number: SC B4 87 77 Site and layer: Santa Catalina II

Typology: unworked
ZooMS ID: Fin whale

14C date: MUSE20066, 12670 ± 60 BP, 15204-14850 cal BP
(photograph taken after sampling)



Supplementary Figure 72 Study number: SC B6 109 1085 Museum number: SC B6 109 1085 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale

 14 C date: MUSE20038, 13010 ± 50 BP, 15634-15119 cal BP



Supplementary Figure 73 Study number: SC B6 135 24 Museum number: SC B6 135 24 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale ¹⁴C date: MUSE20039, 13060 ± 50 BP, 15699-15177 cal BP



Supplementary Figure 74 Study number: SC B6 151 48 Museum number: SC B6 151 48 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale

¹⁴C date: MUSE20067, 12990 ± 60 BP, 15624-15082 cal BP



Supplementary Figure 75 Study number: SC B6 152 44 Museum number: SC B6 152 44 Site and layer: Santa Catalina III

Typology: unworked
ZooMS ID: Fin whale

14C date: MUSE20068, 13170 ± 60 BP, 15868-15293 cal BP
(photograph taken after sampling)



Supplementary Figure 76 Study number: SC B6 83 67 Museum number: SC B6 83 67 Site and layer: Santa Catalina II

Typology: unworked ZooMS ID: Fin whale

 14 C date: MUSE20036, 12970 ± 50 BP, 15588-15072 cal BP



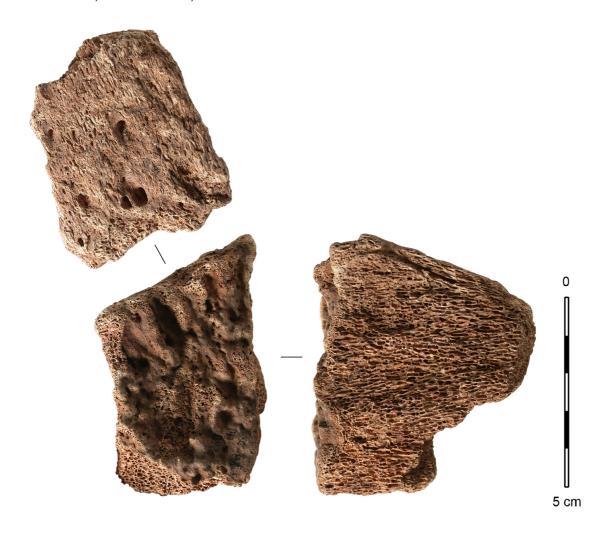
Supplementary Figure 77
Study number: SC B8 124 1180
Museum number: SC B8 124 1180
Site and layer: Santa Catalina III
Typology: unworked
ZooMS ID: Fin whale

14C date: MUSE20041, 13090 ± 50 BP, 15740-15212 cal BP



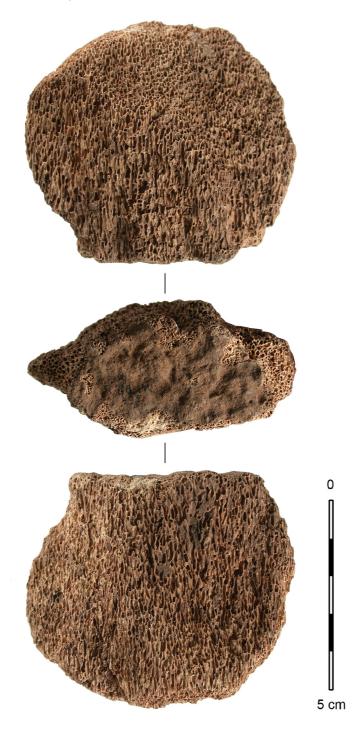
Supplementary Figure 78 Study number: SC B8 143 1392 Museum number: SC B8 143 1392 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale / Right whale / Bowhead whale ¹⁴C date: MUSE20043, 13040 ± 50 BP, 15672-15153 cal BP



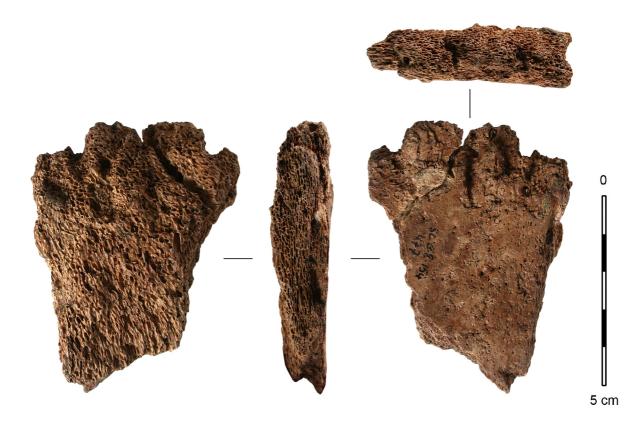
Supplementary Figure 79
Study number: SC B8 144 439
Museum number: SC B8 144 439
Site and layer: Santa Catalina III
Typology: unworked
ZooMS ID: Fin whale

14C date: MUSE20045, 13090 ± 50 BP, 15740-15212 cal BP



Supplementary Figure 80 Study number: SC B8 151 477 Museum number: SC B8 151 477 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale ¹⁴C date: MUSE20047, 13080 ± 50 BP, 15727-15201 cal BP



Supplementary Figure 81 Study number: SC B8 153 130 Museum number: SC B8 153 130 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale

¹⁴C date: MUSE20069, 13010 ± 60 BP, 15648-15106 cal BP

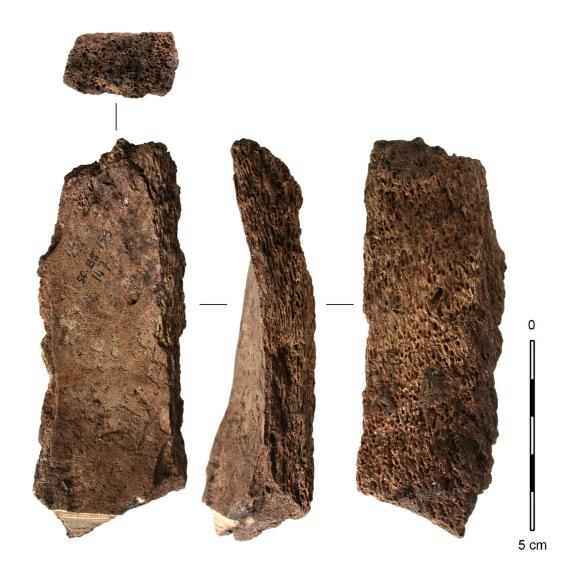
(photograph taken after sampling)



Supplementary Figure 82 Study number: SC B8 153 147 Museum number: SC B8 153 147 Site and layer: Santa Catalina III

Typology: unworked
ZooMS ID: Fin whale

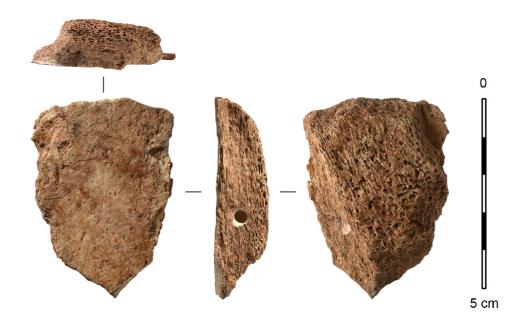
14C date: MUSE20033, 13070 ± 60 BP, 15726-15175 cal BP
(photograph taken after sampling)



Supplementary Figure 83 Study number: SC B8 154 129 Museum number: SC B8 154 129 Site and layer: Santa Catalina III

Typology: unworked
ZooMS ID: Fin whale

14C date: MUSE20071, 13130 ± 60 BP, 15811-15246 cal BP
(photograph taken after sampling)



Supplementary Figure 84 Study number: SC B8 154 150 Museum number: SC B8 154 150 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale / Gray whale / Humpback whale ¹⁴C date: MUSE20048, failed



Supplementary Figure 85 Study number: SC B8 L31G 3932 Museum number: SC B8 L31G 3932 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale ¹⁴C date: none (scale 1:2)



Supplementary Figure 86 Study number: SC B8 L31H 2251 Museum number: SC B8 L31H 2251

Site and layer: Santa Catalina III
Typology: unworked
ZooMS ID: Fin whale

14C date: none

(photograph taken after sampling)



Supplementary Figure 87 Study number: SC C4 L29 229 Museum number: SC C4 L29 229 Site and layer: Santa Catalina II

Typology: blank?
ZooMS ID: Right whale / Bowhead whale

14C date: none



Supplementary Figure 88

Study number: SC C6 159 1 Museum number: SC C6 159 1 Site and layer: Santa Catalina III

Typology: unworked ZooMS ID: Fin whale ¹⁴C date: MUSE20042, 13060 ± 50 BP, 15699-15177 cal BP



Supplementary Figure 89
Study number: SC C8 149 354
Museum number: SC C8 149 354
Site and layer: Santa Catalina III

Typology: unworked
ZooMS ID: Fin whale

14C date: MUSE20074, 13060 ± 50 BP, 15699-15151 cal BP
(photograph taken after sampling)

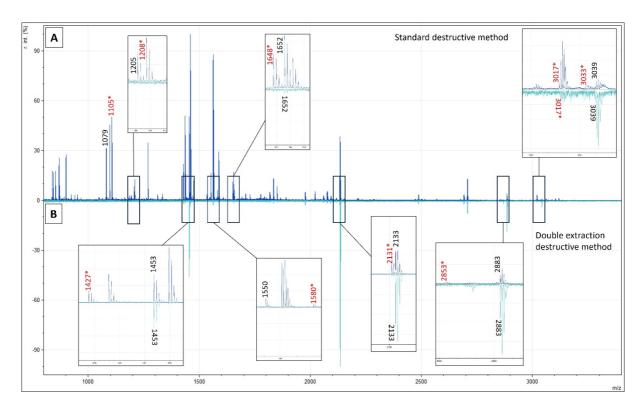


Supplementary Figure 90 Study number: SC C8 158 130 Museum number: SC C8 158 130 Site and layer: Santa Catalina III

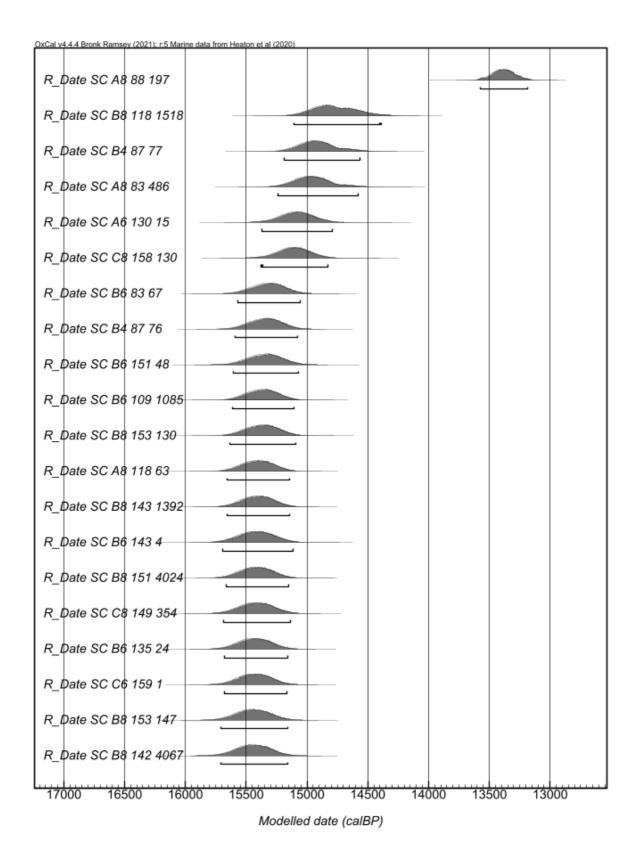
Typology: unworked
ZooMS ID: Fin whale

14C date: MUSE20128, 12810 ± 60 BP, 15390-14844 cal BP
(photograph taken after sampling)

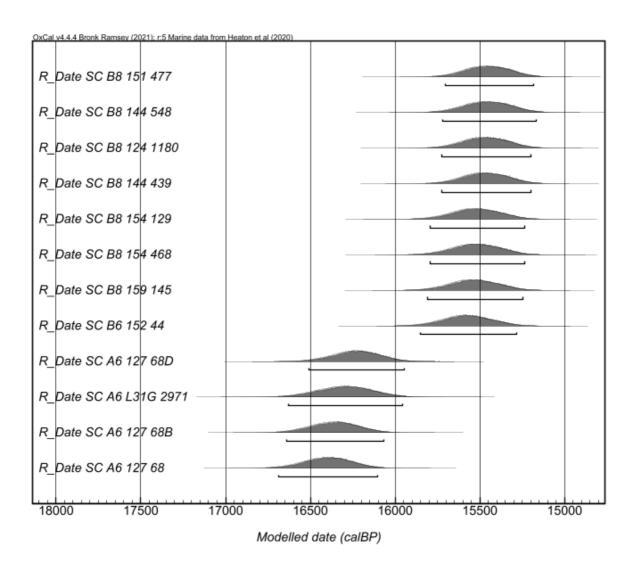




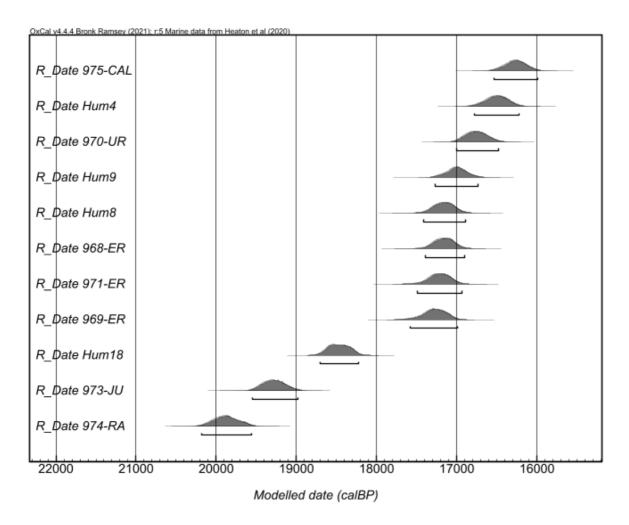
Supplementary Figure 91. MALDI-TOF-MS spectra for a cetacean bone artifact (a wedge, identifier 780, Supplementary Figure 22) from the site of Isturitz excavated by St-Périer. Top spectrum (A) shows the mixed whale and cattle markers obtained when the standard destructive extraction method was performed; lower spectrum (B) shows the whale markers and reduced cattle signal following the double extraction destructive method. Inset images provide detailed views of key peptide markers used for both cetaceans (in this case a sperm whale, black text) and cattle (red text with *). Spectrum intensity has been normalised for comparative purposes.



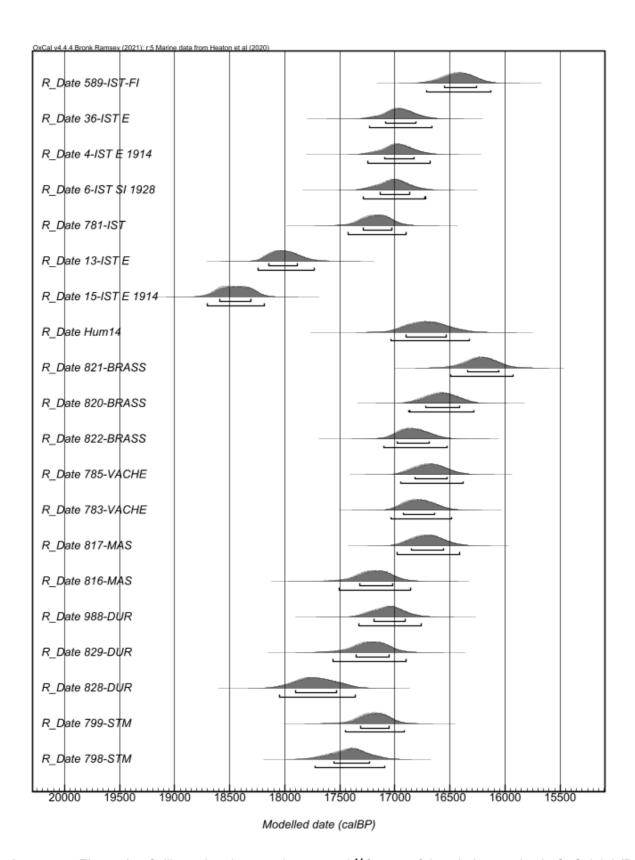
Supplementary Figure 92. Calibrated and reservoir corrected ¹⁴C ages of the whale samples in OxCal 4.4 (Bronk Ramsey 2009), from youngest to oldest. Samples from the site of Santa Catalina (Part I). See Supplementary Data 1 for source data and more information on each sample, including species. See Supplementary Code 1 file for source code.



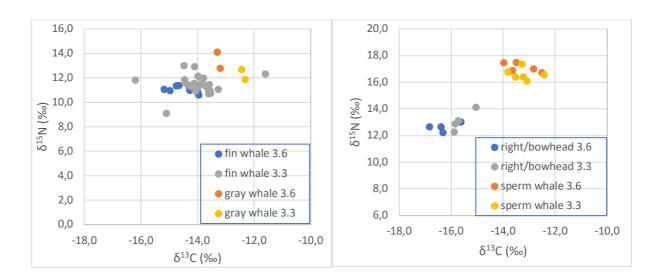
Supplementary Figure 93. Calibrated and reservoir corrected ¹⁴C ages of the whale samples in OxCal 4.4 (Bronk Ramsey 2009), from youngest to oldest. Samples from the site of Santa Catalina (Part II). See Supplementary Data 1 for source data and more information on each sample, including species. See Supplementary Code 1 file for source code.



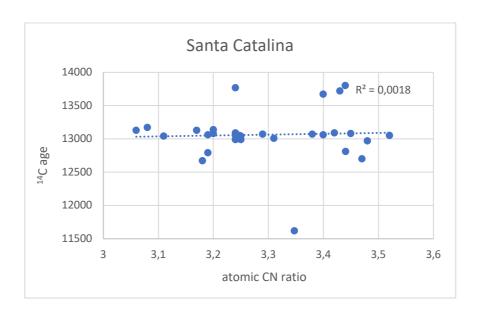
Supplementary Figure 94. Calibrated and reservoir corrected ¹⁴C ages of the whale samples in OxCal 4.4 (Bronk Ramsey 2009), from youngest to oldest. Samples from the Cantabrian sites. See Supplementary Data 1 for source data and more information on each sample, including species. See Supplementary Code 1 file for source code.



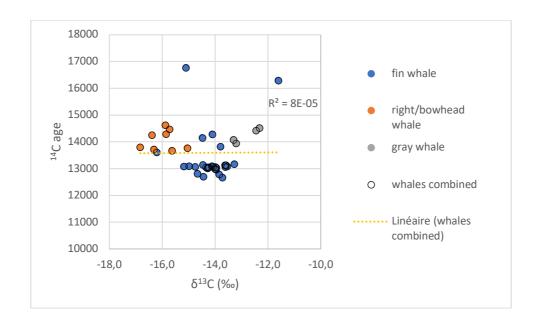
Supplementary Figure 95. Calibrated and reservoir corrected ¹⁴C ages of the whale samples in OxCal 4.4 (Bronk Ramsey 2009), from youngest to oldest. Samples from the French sites. See Supplementary Data 1 for source data and more information on each sample, including species. See Supplementary Code 1 file for source code.



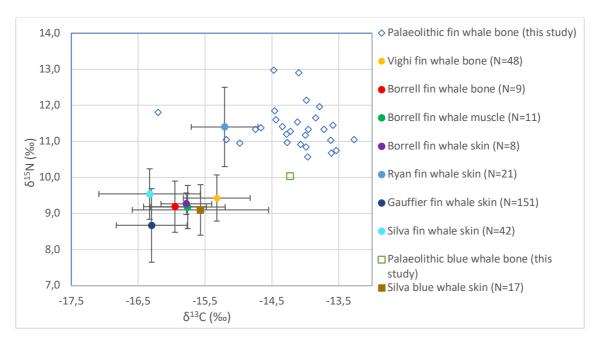
Supplementary Figure 96. δ^{13} C and δ^{15} N ratios of Paleolithic whales (this study), showing the differences between the different atomic CN ratios per species. Number of specimens: fin whale = 31; gray whale = 4; right/bowhead whale = 8; sperm whale = 11. See Supplementary Data 4, tab "PaleoCet material", for source data.



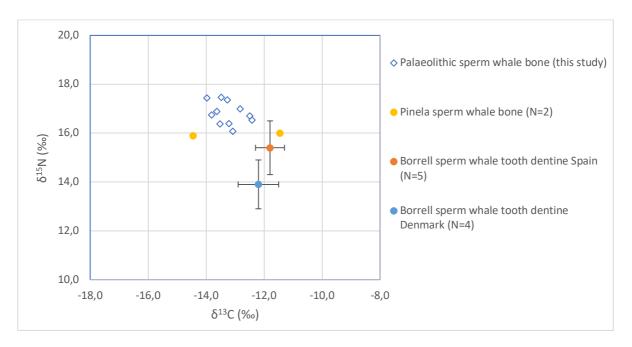
Supplementary Figure 97. ¹⁴C ages plotted against the atomic CN ratio of material from the site of Santa Catalina. A correlation between the two might indicate contamination, which is not the case. Number of specimens = 30. See Supplementary Data 4, tab "PaleoCet material", for source data.



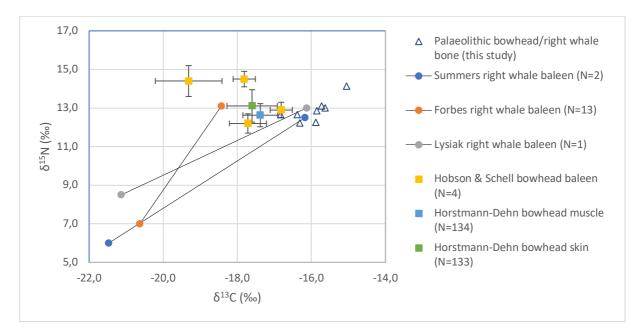
Supplementary Figure 98. Relationship between δ^{13} C ratios and conventional 14 C ages among the analyzed bones of fin, gray and right/bowhead whales. Number of specimens: fin whale = 31; gray whale = 4; right/bowhead whale = 8. See Supplementary Data 4, tab "PaleoCet material", for source data.



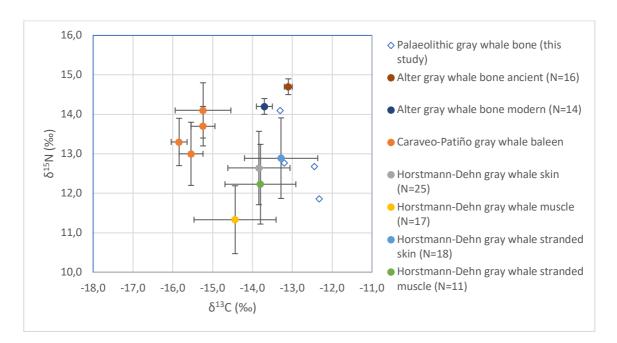
Supplementary Figure 99. δ^{13} C and δ^{15} N ratios from the studied Paleolithic fin whales and blue whale compared with stable isotope values from modern whales from the Atlantic Ocean (after applying Suess effect and, if applicable, tissue corrections)^{1,2,3,4,5}. Whiskers represent errors bars on average values. See Supplementary Data 4 for source data.



Supplementary Figure 100. δ^{13} C and δ^{15} N ratios from the studied Paleolithic sperm whales compared with stable isotope values from modern whales from the Atlantic^{6,1} (after applying Suess effect and, if applicable, tissue corrections). Whiskers represent errors bars on average values. See Supplementary Data 4 for source data.



Supplementary Figure 101. δ^{13} C and δ^{15} N ratios from the studied Paleolithic bowhead/north Atlantic right whales compared with stable isotope values from modern whales from the Atlantic Ocean (right whales)^{7,8,9} and Canadian Arctic (bowhead whales)^{10,11} (after applying Suess effect and, if applicable, tissue corrections). Whiskers represent errors bars on average values, while values from Summers, Forbes and Lysiak present the observed range, connecting the lowest and highest values with a thin line (averages were not available). See Supplementary Data 4 for source data.



Supplementary Figure 102. δ^{13} C and δ^{15} N ratios from the studied Paleolithic gray whales compared with stable isotope values from ancient gray whales ¹², modern whales from the Pacific Ocean ^{13,11} (after applying Suess effect and, if applicable, tissue corrections). Whiskers represent errors bars on average values. See Supplementary Data 4 for source data.

Supplementary Discussion: Comparing stable isotope signatures in Paleolithic versus modern whales

We compared the stable isotope ratios obtained for samples of Paleolithic whales in our dataset with ratios reported in the literature for modern counterparts. Teixeira et al.¹⁴ outline useful guidelines for stable isotope analysis in cetacean research, underlining especially the difficulties when comparing data obtained using different tissues (muscle/skin/baleen/bone), the need for a Suess effect correction, tissue pretreatment (lipid removal), and considering intra- and inter-individual variation. We discuss below how we took into account each of these points.

Tissue corrections

Numerous previous studies applied stable isotope analysis to modern whale tissues, although typically on muscle, skin or baleen. A tissue correction is needed for making these values comparable with bone collagen stable isotope data. Schoeninger and Moore¹⁵ report that the offset between muscle and bone collagen in $\delta^{13}C$ ratios is circa $2\%^{16,17}$. Fischer et al.¹⁸ analyzed carbon and nitrogen stable isotope ratios on bone collagen and flesh of modern fish in Denmark, finding flesh-bone offsets of 2.1 ± 0.6% for $\delta^{13}C$ ratios and -0.6 ± 0.4% for $\delta^{15}N$ ratios. Borrell et al.¹ analyzed the stable isotope ratios of various tissues from fin whales caught between 1983 and 1985 from the Caneliñas whaling station off the coast of Northwest Spain. For bone protein from 9 individuals they found averages of $\delta^{13}C$ = -16.46 ± 0.47% and $\delta^{15}N$ = 9.19 ± 0.71%, and for muscle from 11 individuals of $\delta^{13}C$ = -18.27 ± 0.56% and $\delta^{15}N$ = 9.88 ± 0.58%, and skin values of $\delta^{13}C$ = -18.29 ± 0.38% and $\delta^{15}N$ = 9.97 ± 0.30%. This gives a muscle-bone offset of 1.81% for $\delta^{13}C$ ratios and -0.69 for $\delta^{15}N$ ratios and for skin-bone 1.83% for $\delta^{13}C$ ratios and -0.79 for $\delta^{15}N$ ratios. Based on these values from the literature, we opted for the corrections for both muscle-bone and skin-bone of 2% for $\delta^{13}C$ ratios and of -0.7% for $\delta^{15}N$ ratios.

Baleen is constructed of highly mineralized keratin¹⁹, which is similar to bone collagen or dentine in composition, and therefore we considered that results of stable isotope analyses from baleen are directly comparable to those obtained from bone. While bone remodels and provides an average of many years, baleen plates grow continuously at the base whilst being eroded away at the tip²⁰. Baleen tissue thus represents an archive of information about the animal's life history from the last 2 (gray whale¹³) to 20 years (bowhead whale²¹), depending on species, baleen length and growth rates, making it a very useful tissue to examine changes in foraging through fine time scales such as seasons.

Suess Effect correction

Comparing modern samples with archaeological or historic samples requires a correction to the δ^{13} C ratios for the Suess Effect, which involves the incorporation of largely depleted carbon into the atmosphere through the burning of fossil fuels since the industrial revolution²². We used the SuessR package²³ to calculate the correction needed based on the region and year of sample collection (Supplementary Data 4).

Tissue pretreatment

Bas et al.²⁴ examined the combined effect of delipidation and demineralization on the stable isotope results of sea lion and dusky dolphin bones. Their samples were either left untreated or were treated with a chloroform and methanol solution, followed by demineralization with HCl acid. They found atomic CN ratios above 3.6 for untreated samples, indicating the presence of lipids and/or carbonates. Compared with pretreated samples, all untreated samples had lower δ^{13} C (differences were ranging from 0.3 to 2 %), while this varied for δ^{15} N ratios (differences were ranging from +0.2 to -1.3 %). Das et al.²⁵ had the same findings for δ^{13} C ratios on skin samples. All skin and muscle samples in the literature used for this comparison applied a lipid removal step but no demineralization step. Baleen samples were often subjected to lipid removal (3 out of 5) but not to demineralization. Bone and tooth dentine were treated in a variety of ways in the literature, precluding the application of a systematic correction.

Intra- and inter-individual variation

Smith et al.²⁶ investigated intra-skeletal stable isotope variability in 72 cetacean skeletons from 14 species. They found that for 84.7% of the individuals there was >1% variability in δ^{13} C ratios, and for 55.5% >2% variability. Regarding δ^{15} N ratios, 59.7% of the individuals had >1% and 15.3% >2% variability. The authors assume that variation >1% in δ^{13} C ratios and >2% in δ^{15} N ratios reflects changes in foraging behavior. Borrell et al.¹ analyzed muscle tissues of a large number of individuals (n=65) of fin whale, finding no significant age-related variation (4-65 years), nor significant sex-related differences (n=55 males, n=10 females) in stable isotope values, deducing that this absence of individual variation reflects a consistent use of the same feeding grounds and a similar diet (almost exclusively krill) by different age groups and sexes. Muscle tissue from bowheads in the eastern Arctic showed no age-related isotopic differences, while their baleen revealed that subadult whales feed slightly more in nearshore waters, resulting in lower δ^{13} C ratios, compared with adults in deeper waters²⁷. A significant negative correlation was found between age and δ^{15} N ratios for bowheads, with fetuses and juveniles having higher δ^{15} N ratios than adults, which is most likely due to milk consumption¹¹. It was not possible to take intra- or inter-individual stable isotope variability into account in our analysis.

Fin whales

We contrasted stable isotope ratios obtained from our Paleolithic samples of fin whales (n = 30) with values obtained from modern fin whales in the eastern North Atlantic (after applying Suess effect corrections and, when necessary, tissue corrections) originating from bone samples (9 individuals 1 + 48 individuals 3), muscle samples (11 individuals 1) and skin samples (8 individuals 1 + 21 individuals 2 + 42 individuals 4 + 151 individuals 5).

Fin whales in our sample generally display high $\delta^{15}N$ ratios when compared with their modern counterparts (Supplementary Figure 99), indicating that they occupied on average a higher trophic level than today's whales²⁸, or that the δ^{15} N baseline has significantly changed over time. When compared with the other whale species in our study, modern fin whales are considered a relative generalist predator. In the North Atlantic, their diet is dominated by zooplankton (including multiple euphausiid species) but can also include non-negligible contributions of small pelagic fishes (capelin, Mallotus villosus; Atlantic herring, Clupea harengus; sandlance, Ammodytes spp.²⁹). Feeding strategies can vary across populations, among individuals of the same population, and over time in response to environmental change. For example, an analysis of stable isotope ratios in skin and blubber of 99 fin whales in the Saint Lawrence Estuary over 1998-2006²⁹ found that the population was initially dominated by two types of specialists (individuals focused either on krill or on lipid-rich pelagic fishes). It then shifted toward one composed either of krill specialists or (for about 66% of the individuals) true generalists feeding on various zooplankton and fish prey, a change that coincided with a decrease in biomass of their predominant prey, Arctic krill (*Thysanoessa* spp.). Previous studies also found variation in $\delta^{15}N$ ratios in baleen and muscle samples that reflect seasonal variation in feeding strategies of these highly migratory species: Aguilar et al. 30 (using part of the same dataset as Borrell et al. but only looking at males) found higher δ^{15} N ratios during periods of intense feeding (at high latitudes), that decreased during fasting (migration and calving seasons), whereas Rita et al.³¹ found that δ¹⁵N ratios were higher in winter than in summer. Stable isotope ratios obtained from bones reflect the integration of feeding strategies over years. Accordingly, the relatively high $\delta^{15}N$ ratios in Paleolithic samples could indicate that on average fin whales' diets integrated relatively high fractions of fish when compared to today's whales, which may have been related to the different environmental circumstances.

The Paleolithic whales also exhibit high $\delta^{13}C$ ratios when compared with modern counterparts (Supplementary Figure 99). $\delta^{13}C$ ratios in marine waters vary geographically, with a tendency to decline from coastal and benthic areas to offshore and pelagic areas, and towards polar latitudes³². The higher $\delta^{13}C$ ratios in ancient whales may thus indicate a tendency to rely more on coastal and/or on more tropical and subtropical areas than today's populations.

Blue whales

We contrasted stable isotope ratios of the single blue whale bone sample in our study with skin from 17 blue whale individuals analyzed by Silva et al.⁴, corresponding to blue whales migrating through the Azores and likely reflecting winter and spring feeding grounds in the Northwest African upwelling and pelagic tropical/subtropical regions. The ancient blue whale shows higher δ 13C and δ 15N values than its modern counterparts, and isotopically appears to fall in between the modern and ancient fin whales (Supplementary Figure 99). Modern blue whales are considered dietary specialists, feeding almost exclusively on krill (*Meganyctiphanes norvegica*, *Thysanoessa raschii* and *T. inermis*)⁴. They mostly feed in high-latitude summer grounds, and fast during migration to and from low latitude winter calving areas, but there is evidence of year-round feeding³³.

Sperm whales

We contrasted stable isotope ratios obtained from our Paleolithic bone samples of sperm whales (n = 11) with values obtained from modern sperm whales from the Atlantic (2 bone samples 6 + 9 tooth dentine samples 1) after applying tissue and Suess effect corrections.

We found overlapping ranges of δ^{13} C ratios between modern and ancient whales, with the latter falling on the higher end of the δ^{15} N ratios range in relation to the former (Supplementary Figure 100). Sperm whales are dominant predators of the mesopelagic ocean, preying on many of the larger meso- and bathy-pelagic species of the deep ocean, including cephalopods and fish³⁴. Modern sperm whales in the North Atlantic are highly structured by sex and age, with females and young whales found in tropical and temperate waters whereas older males progressively move towards higher latitudes (migrating periodically southwards for reproduction)^{34,35}. Both males and females are currently found off NW Spain³⁵, but it is plausible that in the cooler Paleolithic oceans the area would have been dominated by older males and this could perhaps explain the higher δ^{15} N ratios in Paleolithic samples. Indeed, previous studies found that δ^{15} N ratios (measured in tooth dentine) tend to increase with age in males, attributed to the increasing strength and diving capacity that individuals acquire when growing old, which allow them to progressively feed on bigger prey (mostly giant squids) at higher trophic levels³⁵. In contrast, an opposite trend (or no age effect) has been observed for females, possibly explained by a tendency to reduce the depth of dives (thus a shift to shallower and smaller prey) during pregnancy and lactation³⁵.

Right/bowhead whales

As we were unable to distinguish between bowhead whale and North Atlantic right whale through ZooMS, we compared the stable isotope signatures of Paleolithic samples identified as one of these species (n=8) with those of modern North Atlantic right whales (baleen of two individuals analyzed by Summers et al.⁷; 13 by Forbes et al.⁹; and one by Lysiak et al.⁸) and bowheads from the Canadian Artic (baleen from 4 individuals from Hobson and Schell¹⁰; muscle from 134 individuals and skin from 133 individuals from Horstmann-Dehn et al.¹²).

Modern bowhead whales and right whales are specialists that feed mainly on calanoid copepod species (most commonly, and respectively, *Calanus hyperboreus* and *Calanus finmarchicus*), but they can also feed on other mesozooplancton species at a higher trophic level (e.g. mysids, euphausiids, chaetognaths)^{34,36}. Due to their distinct feeding habitats at certain latitudes, the species' overlap in δ^{13} C ratios is somewhat unexpected.

Modern right whales tend to have overall lower values of $\delta^{13}C$ and $\delta^{15}N$ ratios than modern bowhead whales, and the stable isotope signatures of Pleistocene bones overlap better with those of the later (Supplementary Figure 101). As the published studies investigating modern right whales observed stable isotopic ranges rather than averages with standard deviations, these are displayed here as such (Supplementary Figure 101). It is important to note that these ranges^{7,8,9} cover the stable isotope value extremes (winter vs. summer feeding) and when averaged, they would most likely display lower $\delta^{13}C$ and $\delta^{15}N$ ratios compared with the bowhead data. Despite the partial overlap between the modern samples of both species precluding a more certain species identification of the Paleolithic bone samples, the ancient samples appear more likely to be bowheads than right whales. Although fin whales are often known to forage at a slightly higher trophic level relative to Balaenidae, when comparing modern fin whales (Supplementary Figure 99) and modern bowhead and North Atlantic right whales (Supplementary Figure 101), we found that fin whales also have lower $\delta^{15}N$ ratios than bowhead whales and a similar range to right whales. This pattern is not only evident among ancie

nt whale samples but also in modern whales and can probably be explained by their different migration styles (coastal vs open ocean) and summer feeding grounds characterized by different baseline δ^{15} N values.

Gray whales

As gray whales are currently extinct in the Atlantic, we could only compare our ancient specimens (n=4) with ancient gray whale from the Pacific (16 bone samples from 150-2690 yr BP¹²) and modern gray whales from the Pacific (14 bone samples¹², baleen from 4 individuals¹³, 25 skin and 17 muscle samples from Alaskan individuals, as well as 18 skin and 11 muscle samples from individuals stranded in California¹¹) (Supplementary Figure 102). Any differences in δ^{13} C ratios may be due to the large geographic distance, although the δ^{15} N ratios overlap considerably, potentially indicating similar foraging preferences. Modern Pacific gray whales are highly coastal, feeding in shallow Artic and subarctic benthic environments, primarily on tube-dwelling ampeliscid amphipods, polychaete worms and bivalves³⁴.

Supplementary references: references cited in the captions of Supplementary Figures 99 through 102, or in the Supplementary Discussion.

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