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Inside baleen: Exceptional microstructure preservation in a late Miocene whale skeleton from Peru

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ABSTRACT

Exceptionally preserved delicate baleen microstructures have been found in association with the skeleton of a late Miocene balaenopteroid whale in a dolomite concretion of the Pisco Formation, Peru. Microanalytical data (scanning electron microscopy, electron probe microanalysis, X-ray diffraction) on fossil baleen are

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provided and the results are discussed in terms of their taphonomic and paleoecological implications. Baleen fossilization modes at this site include molding of plates and tubules, and phosphatization. A rapid formation of the concretion was fundamental for fossilization. We suggest that the whale foundered in a soft sediment chemically favorable to rapid dolomite precipitation, allowing the preservation of delicate structures. Morphometric considerations on the baleen plates and bristles coupled with the reconstructed calcification of the latter permit speculation on the trophic preferences of this balaenopteroid whale: the densely-spaced plates and the fine and calcified bristles provide evidence for feeding on small-sized plankton, as the modern sei whale.

INTRODUCTION

Despite the long and relatively diversified fossil record of baleen-bearing whales (originating ca 34 Ma ago; see Deméré et al., 2008; Marx and Fordyce, 2015), the preservation of baleen structures in situ is extremely rare (e.g., Packard, 1946; Barnes et al., 1987). This sparse fossil record is due to the more rapid decomposition of baleen (formed of keratin and minor hydroxyapatite, Fig. 1A) as compared to bone, and to the detachment of baleen racks from maxillae following the decay of epithelial tissues. Nevertheless, in the Mio-Pliocene Pisco Formation (Peru), fossilized baleen racks have been described associated to several dozens of whale skeletons, strengthening the identification of the Pisco fossil deposits as a Lagerstätte (Pilleri and Pilleri, 1989; Brand et al., 2004; Esperante et al., 2008; Bisconti, 2012).

We describe the exceptional finding of a fossil balaenopteroid whale skeleton with preserved baleen in a dolomite concretion from the Pisco Fm, and interpret the fossil structures through a microanalytical study of the remains, aimed at (i) understanding the

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modes of fossilization and (ii) reconstructing the original characteristics of the baleen. Furthermore, our observations allow us to infer the trophic habits of the studied fossil balaenopteroid.

FIELD DESCRIPTION

The 200 m-thick section of the Pisco Fm exposed at Cerro Colorado (CC hereafter, Fig. 1B) has been recently assigned to the early late Miocene (Tortonian, ca 9 Ma) based on new biostratigraphic and tephrochronological data (Di Celma et al., 2016). The CC site is overwhelmingly rich in marine fossil vertebrates, including mysticetes as well as the holotype of the giant raptorial sperm whale *Livyatan melvillei* (Bianucci et al., 2010, 2016, Lambert et al., 2010, 2015, Collareta et al., 2015).

In September 2014, a partial skeleton of a mysticete whale was discovered (14°21′13.4”S – 75°53′0.1”W) embedded within a layer of diatomaceous silts in the upper portion of the Pisco Fm lower allomember (DR1). The skeleton (M1 in the CC fossil vertebrates map of Bianucci et al., 2016) consists of the skull, both mandibles, both baleen racks (Fig. 1C, D), and portions of the postcranial skeleton (vertebrae, ribs, and forelimbs) enclosed in a hard dolomitic concretion. Despite weathering having caused this concretion to break into several pieces, observations on the parts still preserved in situ indicate that M1 settled and fossilized dorsal-side up. The degree of articulation of M1 is remarkably high; there is no evidence of diagenetic compaction and, except for a moderate degree of recent weathering on exposed bony surfaces, its preservation is excellent. Observations and measurements in the field (DR2) allowed referring M1 to a young adult of the undescribed middle-sized balaenopteroid species recognized at CC by Collareta et al. (2015).

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The right baleen rack is preserved between the medial surface of the right mandible and the right ventrolateral margin of the vomer (Fig. 1D). This rack is apparently complete and fully articulated; it is composed of 2–5 mm thick and regularly spaced plate-like structures, with the spaces in between encrusted by gypsum. We counted 20 plates in a length of 10 cm measured parallel to the long axis of the skull. The baleen rack is not preserved in life position; its base is detached from the lateral margin of the palate and is located along the ventral maxilla-vomer suture, due to a counterclockwise (in posterior view) rotation of ca 90°, exposing the ventral end of the plates near the labial edge of the maxilla. Anterior and posterior surfaces of the plates present thin, parallel grooves, rarely overlapping (Fig. 1E). Light-colored, string-like structures along the medial face of the mandible strongly resemble bristle remains (Fig. 1F, G).

The left baleen rack is associated to the ventral surface of the left maxilla (Fig. 1C). Based on the location of the supposed bristles near the medial edge of the maxilla, we hypothesize that, after deposition, the baleen plates rotated under the weight of the head of the mysticete (thus causing the bristles to touch the palatal face of the maxilla until its suture with the vomer).

Small pieces of the concretion with plate-like and bristle-like structures were sampled from fragments crumbled by natural erosion and deposited in the collection of the Museo de Historia Natural de la Universidad Nacional Mayor de San Marcos (MUSM), in Lima (collection number MUSM 3238). Some were prepared for scanning electron microscopy (SEM), electron probe microanalysis (EPMA), and X-ray diffraction analyses (methodological details in DR3).

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MICROANALYTICAL RESULTS

The grooves along the surfaces of the plate-like structures are < 100 micron deep and have a semicircular cross section. Their morphology and position suggest that they are natural casts of baleen tubules. The plate-like structures do not show any internal circular feature reminiscent of tubules, and consist of terrigenous particles and diatom frustules in a dolomite cement (Fig. 2A).

The sediment containing bristles is constituted by diatom frustules thoroughly cemented by microcrystalline dolomite. The diatom frustules are slightly dislocated but not deformed by diagenetic compression (Fig. 2B), and show perfect preservation of silica ultrastructures (DR3). The sediment contains biogenic phosphatic fragments carved by microborings, the latter being filled by iron-bearing framboids (Fig. 2C).

When freed from the enclosing sediment, the objects we interpret as fragments of bristles consist of dolomite rods wrapped in an exfoliating material strongly resembling the arrangement of the epithelial cells of bristles (Pfeiffer, 1992) (Fig. 2D, F). Upon sectioning, the bristles show the inner lumen completely filled with dolomite, and the "walls" consisting of Ca-phosphate with layered structure (Fig. 2F). The peaks characteristic of fluorapatite are visible in X-ray diffraction spectra of the fossil bristle material, and the major element composition determined by EPMA corresponds to F-bearing apatite (see DR3 for data). As in the diatom frustules, the bristles are fragmented and displaced but the section of tubules is not deformed by compaction.

DISCUSSION

The plate-like structures of the fossil baleen racks are composed of a sediment framework cemented by dolomite and display casts of the baleen tubules only along the

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outer surfaces. This confirms that these structures represent natural casts of the spaces between adjacent baleen plates in a rack, preserved thanks to the formation of the concretion. Their sediment framework indicates that the gaps between adjacent baleen plates - which, in the living whale, are maintained because the plates are regularly set within a soft tissue made of keratin (Fudge et al., 2009; Young et al., 2015) - were filled by sediment as soon as the whale settled along the sea bottom, before the start of keratin decay. Such a process would only be possible if the sea bottom was a soft sediment allowing the carcass to sink (Martill, 1993; Gariboldi et al., 2015), as supported by the high degree of articulation of the skeleton.

But, if the keratinous plates decayed away, how did the keratinous tubules survive long enough to leave their permanent imprint in the concretion? The answer comes from the free bristles we found inside the concretion. The 3D morphology of the bristle fragments is incredibly regular thanks to dolomite filling the medullary lumen (Fig. 2). Although the phosphatic material forming what remains of the cuticle of the tubules is now soft and erodible, the concretion holds a perfect imprint of this cuticle. This suggests that the tubules underwent a different decay and fossilization process than the keratin matrix of the baleen plates. Szewciw et al. (2010) showed that there is an uneven distribution of hydroxyapatite in the baleen of extant mysticetes, with strong interspecific differences: intertubular calcification prevails in the minke whale (*Balaenoptera acutorostrata*), whereas tubule calcification prevails in the sei whale (*Balaenoptera borealis*) and the humpback whale (*Megaptera novaeangliae*). The highest measured values of hydroxyapatite are recorded in the bristles of sei whale (14.5% dry weight; Pautard, 1963). Based on these observations, we propose that the baleen of the

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balaenopteroid from CC were characterized by uneven calcification, with a higher concentration of apatite in the tubules. The higher calcification of the tubules may have stiffened them, so that they persisted even after the keratin of the plate matrix decayed away and left their imprints at the surface of the casts of the plates.

The "walls" of bristles have been replaced by fluorapatite, with a layered arrangement. Despite the porous appearance, it is unlikely that the observed Ca-phosphate is provided only by the original calcium salt crystallites. The fluorapatite could, instead, indicate a process of phosphate precipitation from seawater (phosphatization, e.g., Schiffbauer et al., 2014). The layered arrangement (Fig. 2F), recalling the annular distribution of calcification (Pautard, 1965; Szewciw et al., 2010), permits to infer that the original hydroxyapatite crystallites acted as nucleation sites for mineralization.

The rapid decrease of permeability related to the formation of the concretion is a main preservation factor (McCoy et al., 2015a, b). All evidence suggests early formation of the concretion, before the deformation by compaction of the fragile diatom frustules and before the obliteration by decay of the three-dimensional morphology of keratinous structures (Long and Trinajstic, 2010). However, as indicated by the low degree of phosphatization of bristle walls, the concretion formation may also have impeded further mineralization of the tubule structure.

Both the precipitation of dolomite for the concretion and the mechanism of phosphatization require an alkaline environment. A favorable alkaline environment was most likely induced by the decay of the whale's organic matter, allowing early carbonate precipitation (e.g., Allison, 1988; Yoshida et al., 2015). In the concretion enclosing M1,

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evidence for biomediated sulfate reduction (the relics of pyrite framboids in microborings, Fig. 2C) indicate conditions favorable for dolomite primary precipitation, as proposed for other dolomite concretions hosting vertebrate remains in CC (Gariboldi et al., 2015 and references therein). It cannot be excluded that the whale fell in a soft sediment layer close to the conditions for sulfate reduction, i.e., already rich in decaying organic matter.

Since in extant whales the calcification pattern of tubules is correlated to prey types, with mechanically strong, fine bristles corresponding to smaller prey (Szewciw et al., 2010), we can propose a similar correlation for M1. This is further supported by (i) the mean diameter of the baleen bristles in M1, which is low compared to most extant mysticetes (especially among Balaenopteroidea), and is similar to values reported by Young (2012) for the sei whale and the pygmy right whale (*Caperea marginata*), and by (ii) the relatively high plate density, comparable to densities observed in these two modern species. Both the sei and the pygmy right whale are currently known to feed mostly on small planktonic crustaceans (copepods) (see Young, 2012, and references therein). Interestingly, copepods are also the target prey of the Pacific pilchard *Sardinops*; the latter was abundant along the southern coast of Peru during the early late Miocene and occupied a prominent position in trophic chains of the CC vertebrate fauna (Lambert et al., 2015; Collareta et al., 2015). Therefore, we speculate that the middle-sized balaenopteroid species of CC represented by the specimen M1 fed predominantly on small-sized plankton, and more specifically on copepods.

CONCLUSIONS

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Baleen fossilization modes in a balenopteroid specimen of the Pisco Fm include i) molding of baleen plates in a dolomite concretion, with imprints of the medullary tubules, and of bristles; ii) phosphatization of bristles favored by the same alkaline environment allowing dolomite formation. Possibly favored by sinking of the whale in a soft sediment layer chemically prone to dolomite precipitation, rapid formation of the concretion in respect to decay is testified by the lack of compression of the diatomaceous sediment and by the preservation of the delicate baleen structures.

The preservation of the casts of medullary tubules and bristles requires that decay of the tubules was delayed in respect to decay of the keratin matrix, suggesting a higher calcification of the tubules as compared to the intertubular keratin in the mysticete studied here. Based on the calcification of bristles, their diameter in the lower range of extant mysticetes and the high baleen plate density, we propose that this middle-sized balaenopteroid species fed predominantly on small-sized zooplankton, as the modern sei whale and the pygmy right whale.

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305 FIGURE CAPTIONS
306
307 Figure 1. A. Main baleen features of an extant rorqual, at different scales; tubules
308 diameter is on the order of 0.5 mm (modified and redrawn from Szewciw et al., 2010). B.
309 Location of Cerro Colorado (see detailed map in DR1). C, D. The right (D) and left (C)
310 baleen racks of the balaenopteroid M1 as found in the field, with drawings showing their
311 original position in the whale skull. Dolomite = dolomite concretion. E. Surface of plate-
312 like structure with tubule casts (arrow) in specimen M1. F. Fossil bristles of specimen M1
313 at the end of plates. G. The same as in F, in extant balaenopterid baleen.
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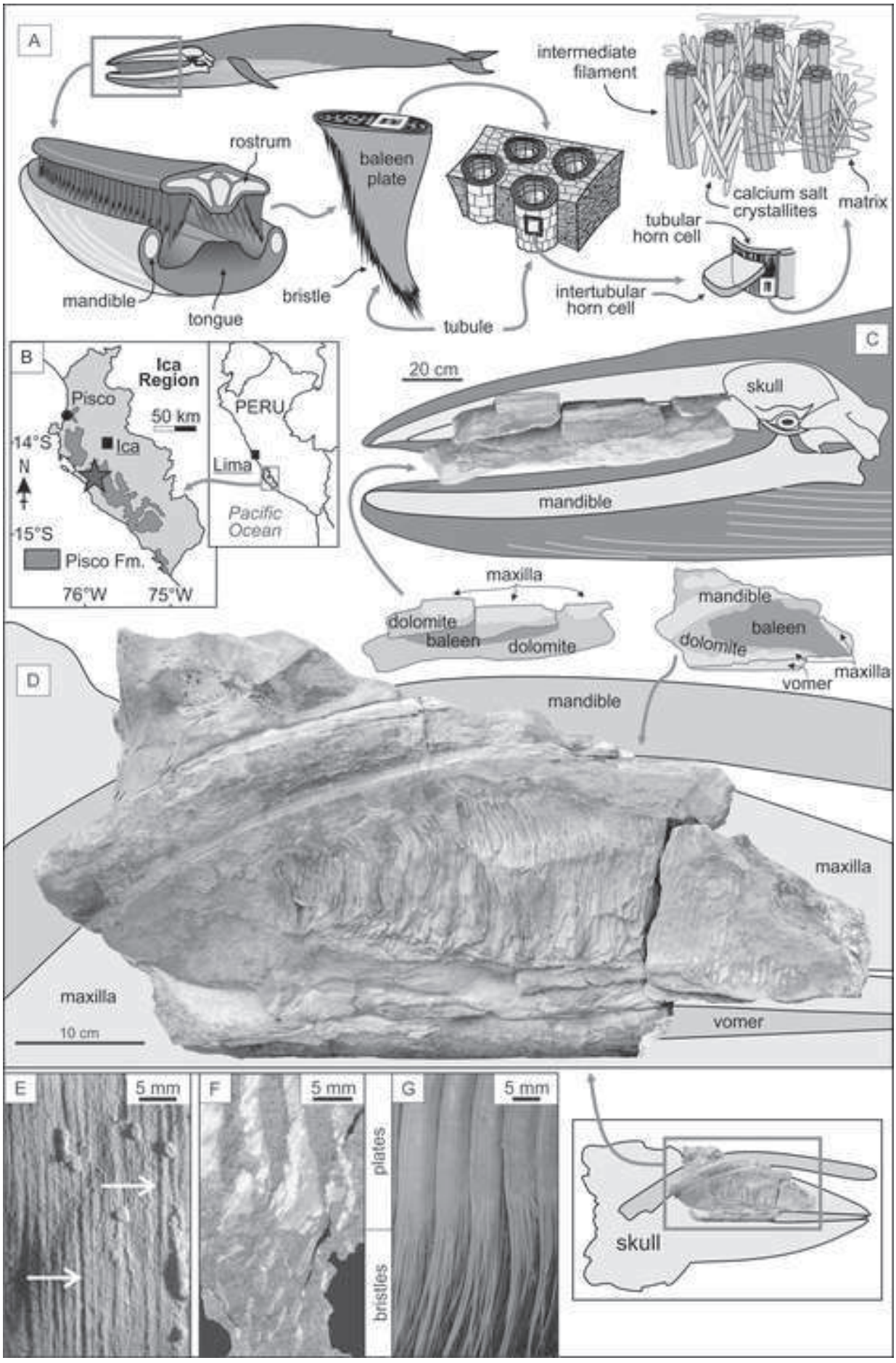
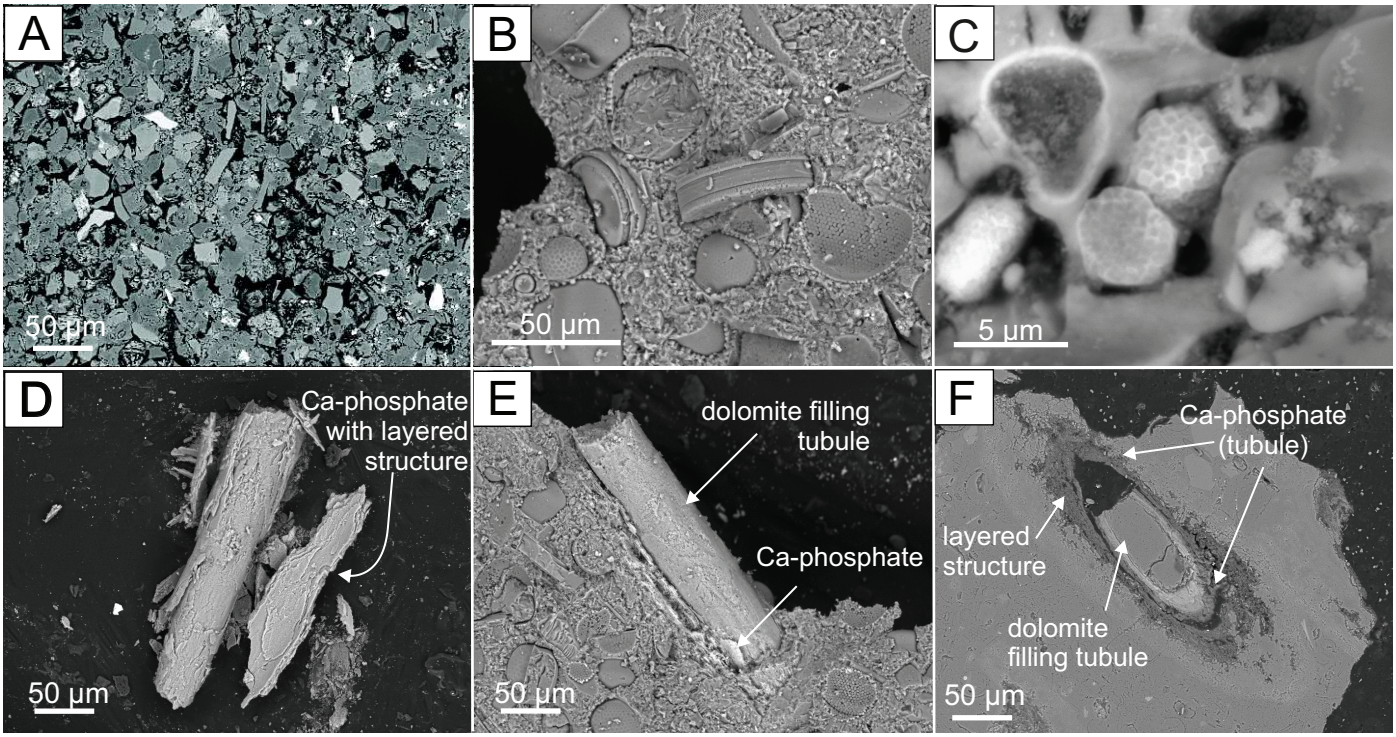
Figure 2. SEM images of the dolomite concretion (A, B), of framboids in microboring of phosphatic fragments (C), of fossil bristle fragments (D, E) and section (F). Backscattered electron images except D and E (secondary electron images).

[1]GSA Data Repository item 201Xxxx, geological map and stratigraphic section, paleontological details, analytical details and data, is available online at



Gioncada et alii, Figure 2



Gioncada et alii, DR1

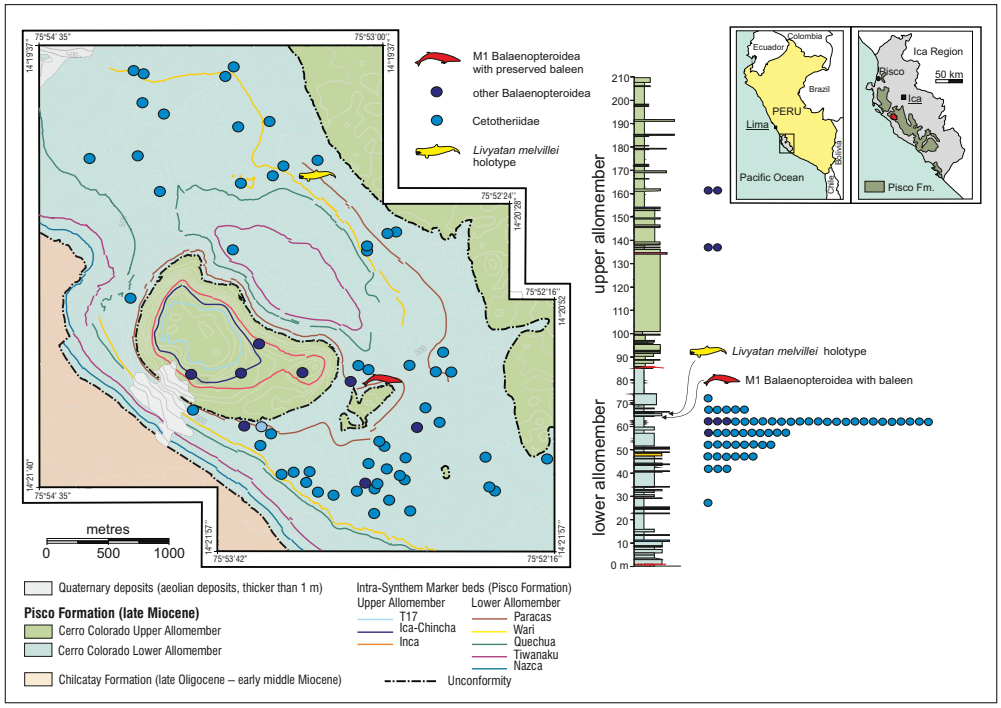


Figure DR1. Cerro Colorado geological map and stratigraphic section modified from Bianucci et al. (2016), showing the distribution of fossil mysticetes and the position of M1. The position of *Lyviatan melvillei* holotype finding is also reported.

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Gioncada et alii, DR2. Additional paleontological observations on the balaenopteroid whale skeleton M1

M1 displays a high degree of articulation, e.g.: both mandibles are still in anatomic connection with the cranium; the forelimbs show perfectly articulated carpal and metacarpal bones; the exposed segment of vertebral column presents articulated vertebrae; the bony thorax (thoracic vertebrae and ribs) preserves its three-dimensional architecture (see figure below).

The skull, mandibles, and ear bones of M1, which are divided into several blocks and fragments, display a typically balaenopteroid outline. A bizygomatic width of ca 96 cm has been estimated for M1. This measurement allows calculating a total body length of ca 8.5 m based on the equation proposed by Lambert et al. (2010). Based on the presence of vertebral epiphyses ankylosed to the relative apophyses, M1 is regarded as a fully grown adult individual; as such, it is here attributed to the undescribed middle-sized (ca 7-9 m long) balaenopteroid species recognized at Cerro Colorado by Collareta et al. (2015).

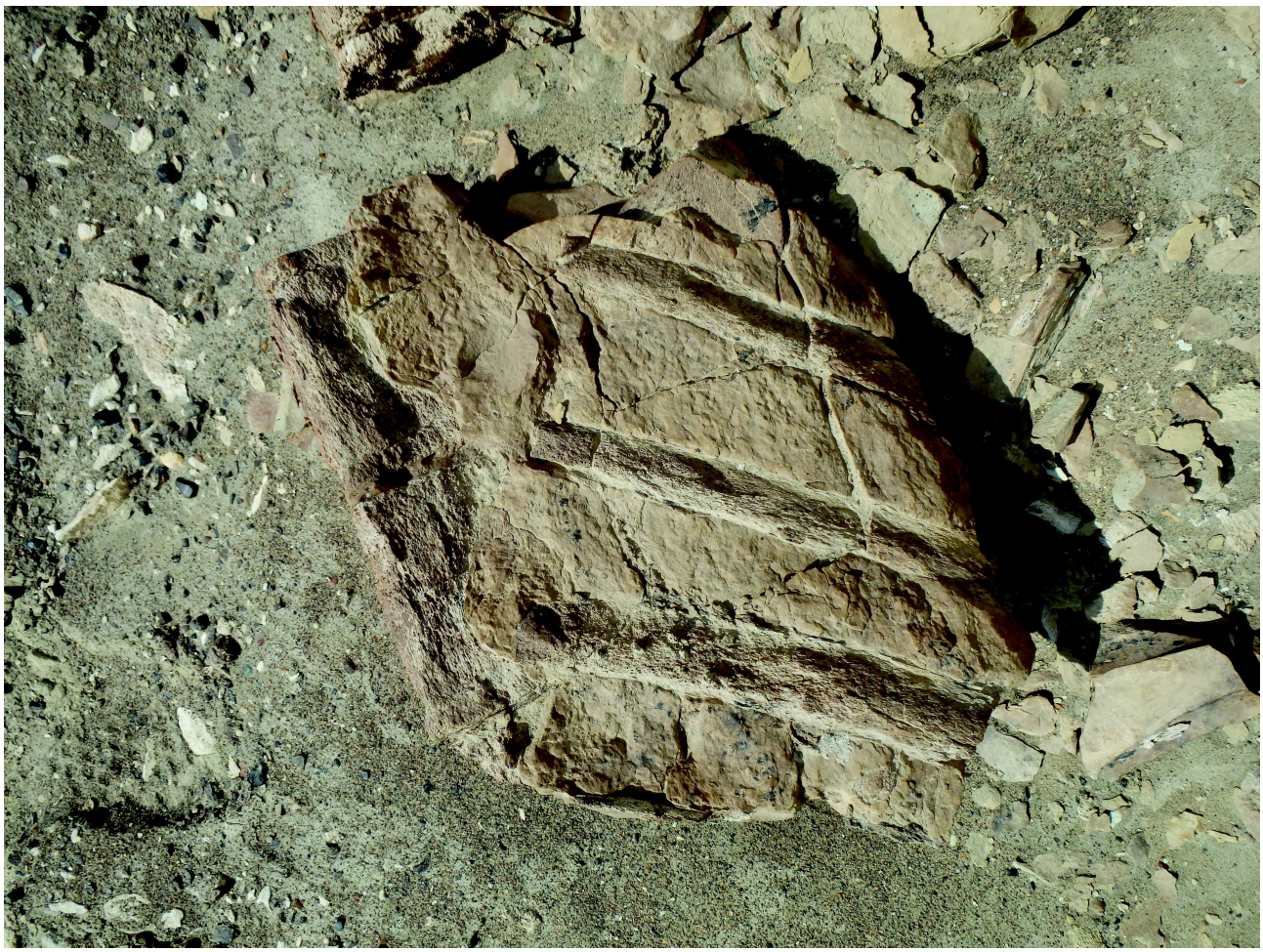

Figure. The bony thorax (thoracic vertebrae and ribs) of M1 preserves its three-dimensional architecture attesting the high degree of articulation of the fossil mysticete skeleton (long dimension of the photo is about 1 m).

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DR3. Material preparation, analytical methods and data

Material preparation and analytical methods

Small pieces of the concretion holding plate-like structures were mounted in resin and sectioned, orthogonally to and along the grooves; one fragment holding bristles was gently crushed to expose the delicate strings. Some phosphatic material was collected from the fossil bristles with a needle and powdered for X-rays diffraction analysis (XRPD). Some fragments were mounted in resin within tube-like holders and polished exposing a section of the string orthogonal to its long axis; others were mounted without resin. The mounts were carbon-coated for scanning electron microscopy with backscattered electrons imaging and energy-dispersive X-ray spectroscopy (SEM-EDS, Philips XL30 SEM equipped with DX4i EDAX microanalysis) and wavelength-dispersive spectroscopy electron probe microanalysis (WDS-EPMA, Cameca SX50 instrument at CNR, Rome, Italy). X-ray powder diffraction (XRPD) data were collected on a Bruker D2 Phaser diffractometer operating at 10 mA and 30 kV, using a flat background-free sample holder and Cu Kα radiation with $\lambda = 1.54178$ Å. Data were processed using the software DIFFRAC.EVA V4.1.

Data

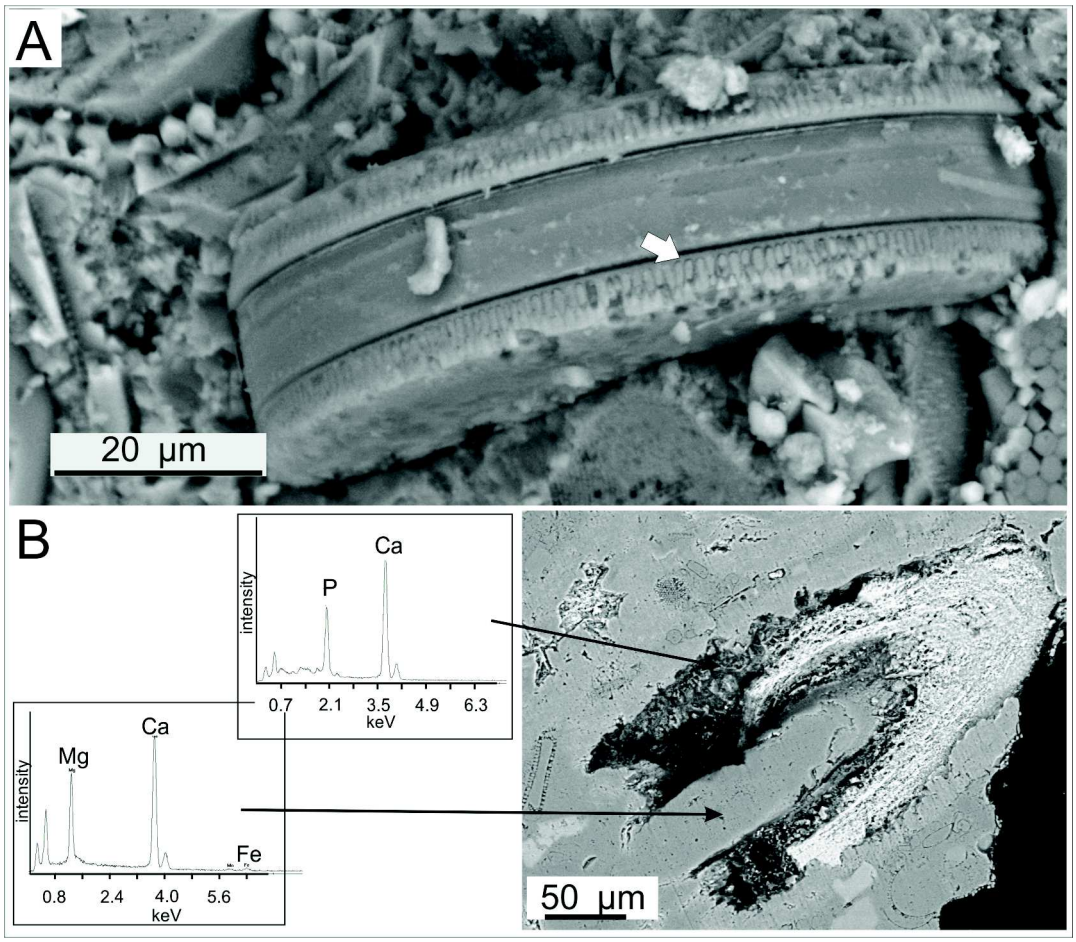
SEM-EDS

Through SEM-EDS analysis, the concretion forming the plates casts and containing fossil bristles was carefully investigated to identify the fossil evidence of tubules and their mineral composition. Analytical conditions were 20 kV accelerating voltage, 5 nA beam current.

The well cemented diatomaceous sediment encasing free bristles (Fig. 2b in the paper) has >95% of diatom frustules and minor terrigenous particles; 95% of the diatom assemblage is made of *Stephanopyxis* spp. and frustule outlines lack any indication of diagenetic compression. In the enlargement here below (A), *Azpeitia* sp. frustule can be recognized; silica is preserved and details of the frustules ultrastructure are still visible; white arrow indicates diamond-shaped areolae on the

mantle and no signs of diagenetic compression are found (SEM-SE). In B, an example of Ca-phosphate and dolomite identification by SEM in a sectioned fossil bristle is shown. The brighter area corresponds to Ca-phosphate permeated by late gypsum (SEM-BSE).

Figure DR3_1

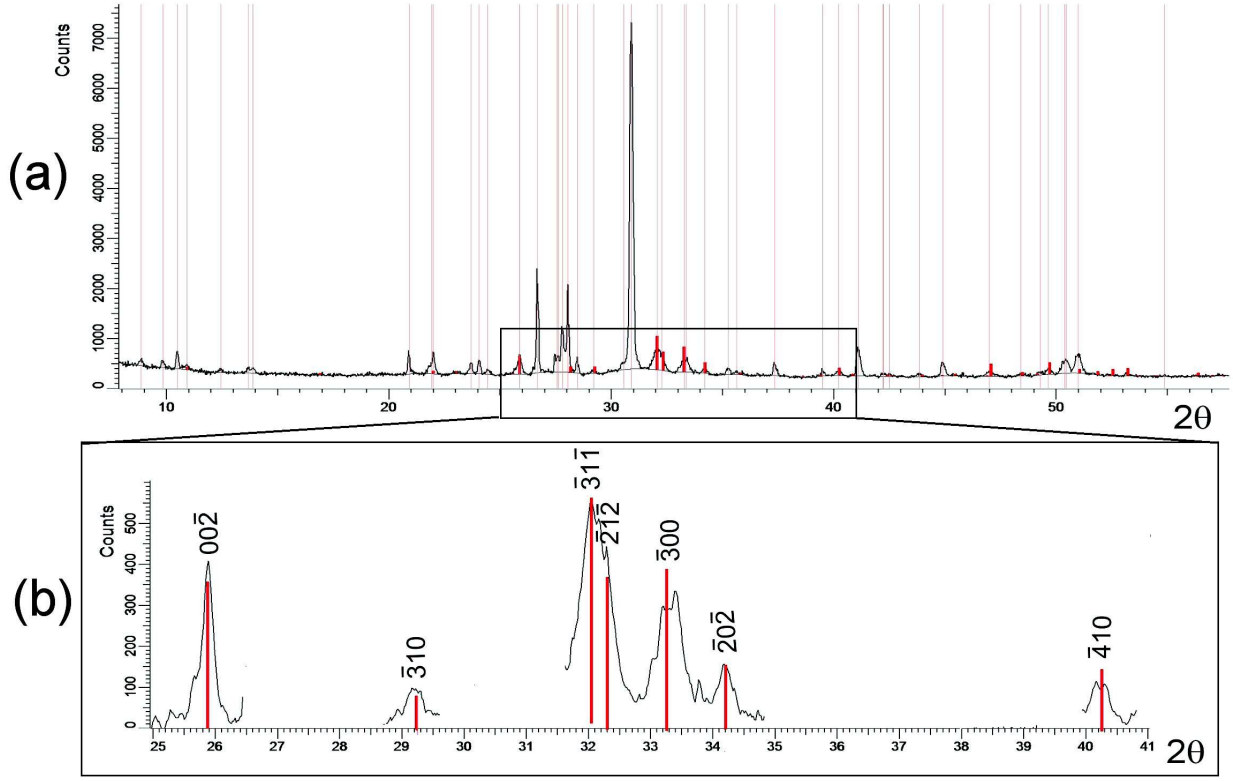


X-rays DIFFRACTION

The sample was manually selected under binoculars. A preliminary X-ray diffraction powder (XRPD) pattern indicated that the occurring main phases were dolomite, gypsum and quartz. The same sample was gently ground in water; after two hours the water was removed and the sample was dried at 80°C in a furnace. The final XRPD pattern is reported in Figure DR3_2(a); the identified phases were dolomite, quartz, minor amounts of orthopyroxenes, amphibole and plagioclase, and, as emphasized in Figure DR3_2(b), apatite-(CaF) (‘fluorapatite’). In the enlarged portion of

the XRPD pattern, only the ‘fluorapatite’ peaks not superimposed to indexed peaks of other phases are reported.

Figure DR3_2



ELECTRON MICROPROBE

EPM analyses of Ca-phosphate of the bristles and of the dolomite are reported in Table DR3_1 (below). The Ca-phosphate analyses give values for the Ca/P atomic ratio which are higher compared to hydroxyapatite composition (Wopenka and Pasteris, 2005), but in the range of published bioapatite bone materials (Li and Pasteris, 2014). The occurrence of low amounts of sulfur in analyses 1-4, joined to the relatively high Ca content, could be due to the pervasive occurrence of gypsum within porosity. We attribute the low totals of Ca-phosphate compared to published EMPA data (Li and Pasteris, 2014) to the imperfect surface obtained when polishing such porous material.

	Ca-phosphate				dolomite			
	1	2	3	4	5	6	7	8
wt%								
SO_3	1.84	1.99	1.48	1.64	0.02	bdl	0.03	0.06
P_2O_5	31.28	31.42	30.38	31.94	0.06	0.09	0.12	0.18
SiO_2	1.34	1.49	1.43	1.77	0.02	bdl	0.06	0.01
Fe_2O_3tot	0.06	0.12	0.05	0.10	0.06	0.02	0.02	0.05
MgO	1.06	1.21	1.15	1.26	21.69	20.79	20.48	21.06
CaO	44.35	44.93	44.35	45.13	31.98	31.29	31.55	31.42
MnO	0.04	0.05	0.05	0.05	0.03	0.03	bdl	0.05
SrO	0.14	0.30	0.16	0.15	0.13	0.15	0.01	0.14
PbO	bdl	bdl	0.03	bdl	0.06	bdl	bdl	0.00
Na_2O	0.90	1.01	0.95	0.91	0.07	0.09	0.04	0.08
F	2.86	2.86	3.14	2.88	0.08	bdl	0.08	0.01
Cl	0.13	0.11	0.20	0.20	0.01	0.02	0.01	0.02
sum	84.01	85.48	83.34	86.02	54.20	52.47	52.40	53.06
Ca/P atomic ratio	1.80	1.81	1.85	1.79				

Table DR3_1. Electron probe WDS analyses of the phosphatic material now forming the bristles (1-4) and of the dolomite cementing the concretion (5-8); bdl: below detection limit; Fe_2O_3tot: all Fe as Fe_2O_3. Analytical conditions: 15kV accelerating voltage, 5 nA beam current. A 10 micron defocused beam was used.

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