




MARINE RESERVOIR EFFECT OF SPERMACETI, A WAX OBTAINED FROM THE HEAD OF THE SPERM WHALE: A FIRST ESTIMATION FROM MUSEUM SPECIMENS

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ABSTRACT. Spermaceti is a waxy substance found in the head cavities of sperm whales (*Physeter macrocephalus* and *P. catodon*). This substance had a variety of commercial applications from the end of the 18th to the beginning of the 20th century, such as candles, soap, cosmetics and other compounds. Spermaceti was also occasionally used as wax for modeling sculptures. In order to date such artworks the marine reservoir effect (MRE) has to be considered. The chemical library of the Muséum national d'Histoire naturelle (Paris, France) contains samples of spermaceti studied by the French chemist M. E. Chevreul (1786–1889) at the beginning of the 19th century. Eight samples of substances preserved in their original containers were ^{14}C dated. According to the whaling practices and the publications of Chevreul, we estimated that the spermaceti samples came from whales caught between 1805 and 1815. AMS ^{14}C dating results are from 550 to 1180 ± 30 BP, R values between 393 and $1023 (\pm 34)$ ^{14}C yr and ΔR between -168 and $504 (\pm 60)$ ^{14}C yr. The values presented here are the first ever obtained for spermaceti. However, being based on museum specimens, further measurements on crude material would be necessary to refine these results.

KEYWORDS: marine reservoir, museum, radiocarbon, sperm whale, spermaceti.

INTRODUCTION

Spermaceti is a waxy substance found in the head cavities of sperm whales (*Physeter macrocephalus* and *P. catodon*) (Figure 1a). After extraction from sperm oil, spermaceti forms brilliant white, oily crystals that were used as an ingredient in a variety of commercial applications, such as candles, soap, cosmetics, machine oil, leather waterproofing, rust-proofing materials and many pharmaceutical compounds from the end of the 18th to the beginning of the 20th century (Figure 1b). The production of spermaceti candles was responsible for an increase in the whaling industry in the mid-18th century, negatively impacting the sperm whale population (Starbuck 1878; Lengellé 1955; Zallen 2019). Spermaceti wax was also used as an art material for modeling sculptures. To establish accurate ^{14}C dates for artworks made with this wax (Regert et al. 2005), such as the Flora bust of the Bodemuseum in Berlin (Figure 1b) (Reiche et al. 2021), it is essential to determine the impact of marine reservoir effects (MREs) on spermaceti radiocarbon dates (Alves et al. 2018).

For that purpose, well-dated samples of spermaceti were sought at the Muséum National d'Histoire Naturelle (MNHN, Paris, France). The chemical library of the MNHN contains more than 9000 items composed of isolated or synthesized pure molecules and natural extracts of historical products. Among them are spermaceti specimens studied by the French chemist M. E. Chevreul (1786–1889) at the beginning of the 19th century. To determine MRE values for spermaceti, we measured ^{14}C in eight substances preserved in their original containers labeled in French as spermaceti, blanc de baleine or cétine. We present here marine reservoir age estimates (R), calculated as the difference between ^{14}C results and the expected radiocarbon age of spermaceti specimens based on their estimated

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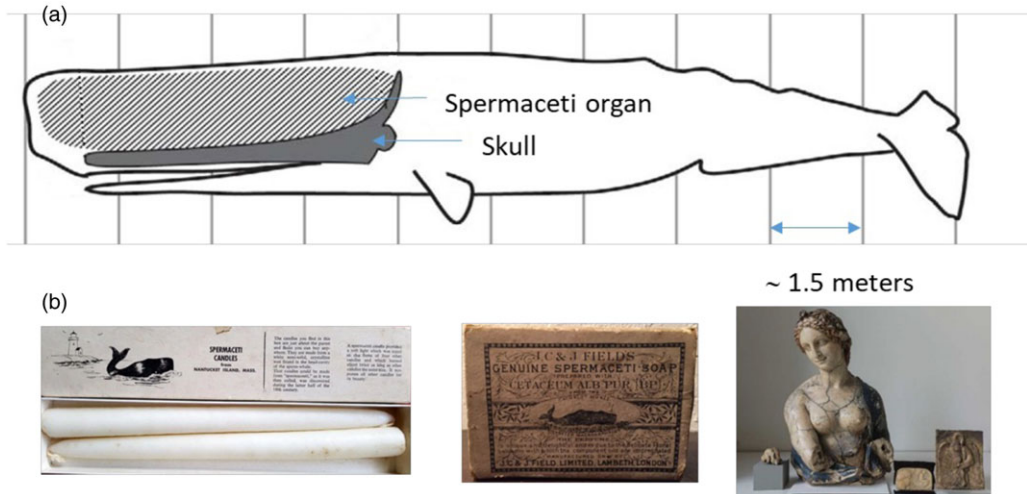


Figure 1 (a) Schematic view of a sperm whale with the location of the spermaceti organ, a cavity filled with almost 2000 L of wax-like liquid called spermaceti (redrawn from Nakamura et al. 2013); (b) examples of ancient manufactured products made with spermaceti: candles, soap and artworks (the “Flora bust” formerly attributed to Leonardo da Vinci [Reiche et al. 2021], Inv. No. 5951, Skulpturensammlung-Museum für Byzantinische Kunst [SBM], Staatliche Museen zu Berlin [SMB]—Stiftung Preußischer Kulturbesitz [SPK] and two objects by Richard Cockle Lucas: “Woman and Winged Woman” Inv. No. SBM Lfd. Nr. 247 and “Leda and the Swan” Alte Nationalgalerie, SMB-SPK, Inv. No. B II 433 © SMB-SPK).

years of collection, and reservoir age offset (ΔR values) estimated from the Marine20 global marine curve (Heaton et al. 2020).

MATERIALS AND METHODS

Sample Description, Estimated Dates of Spermaceti Collection and Whale Death

The Muséum National d’Histoire Naturelle houses spermaceti specimens studied by the French chemist M. E. Chevreul during his work on animal fats. Chevreul reported the properties of spermaceti in his fifth memoir read to the Académie in 1815 (Chevreul 1815). He discovered its composition, principally a cetyl palmitate (ester of cetyl alcohol and palmitic acid, $C_{15}H_{31}COO-C_{16}H_{33}$) that he dubbed “cétine”. Eight glass jars are still preserved in the MNHN collection containing white waxy substances (Figure 2). They are labeled “spermaceti”, “blanc de baleine” (another French term for spermaceti meaning whale white) and “cétine” (cetine). There are two main hypotheses concerning the biological function of the spermaceti organ in sperm whales: buoyancy control or an acoustic role in echolocation (Clark 1970; Koopman 2018).

To estimate ^{14}C ages of these historical spermaceti samples, several parameters were taken into account. The first is linked to the sperm whale itself, its distribution, diet, and metabolism. The second is the duration of different stages occurring after the death of the whale: whaling campaign, spermaceti processing, sale of the final product and Chevreul’s research investigations.

Sperm whales have one of the widest global distributions: they are found in all deep oceans, from the equator to the edge of the Arctic and Antarctic for males. They hunt for food (up to

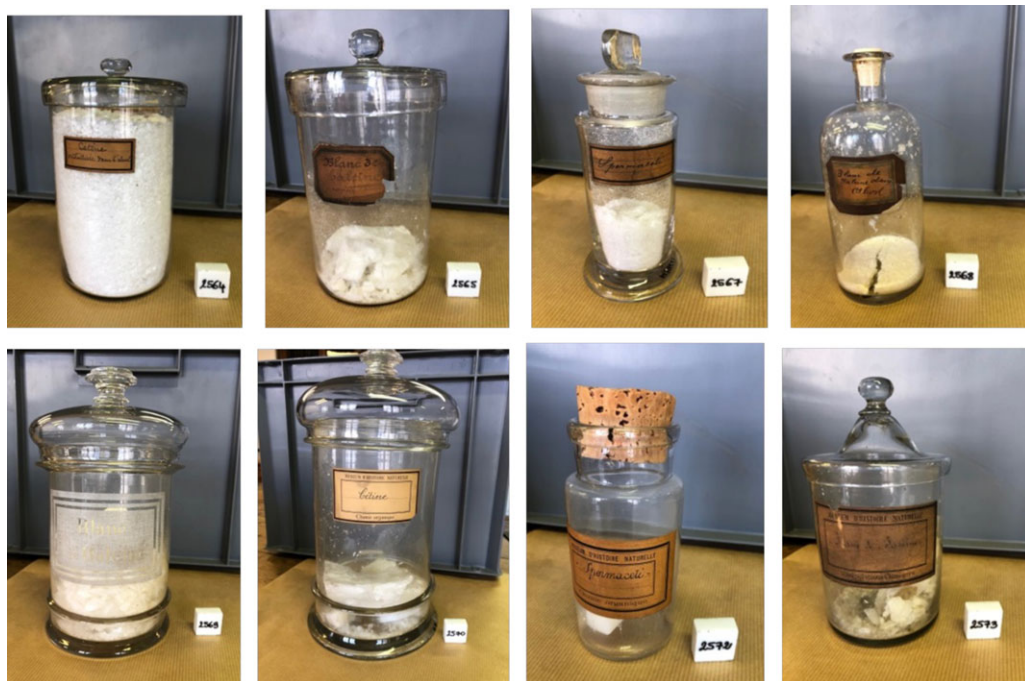


Figure 2 Specimens collected by the French chemist M. E. Chevreul (1786–1889) and preserved at the Muséum National d’Histoire Naturelle (Paris, France) under the reference MNHN-CH-SC. From top left to bottom right: n° 2564 cétine cristallisée dans l’alcool, n°2565 blanc de baleine purifié, n°2567 spermaceti, n°2568 blanc de baleine dans alcool, n°2569 blanc de baleine, n°2570 cétine, n°2572 spermaceti, n°2573 blanc de baleine (see Table 1 for translation of the labels).

one ton per day), mainly cephalopods (squids) (Kawakami 1980), during dives that routinely reach depths of 600–1000 m and regularly more (Clarke 1993). During their long life—up to 60 years—they integrate ^{14}C from various water masses. The turnover rate of spermaceti, which is a liquid composed of wax esters and triglycerides (Wellendorf 1963; Morris 1973) is unknown. However, since the turnover times of fatty acids in human adipose tissue have a half-life in the order of six months to two years (Strawford et al. 2004; Spalding et al. 2017), it might be assumed that spermaceti has a ≤ 10 -yr tissue turnover rate.

The durations of the whaling campaign, spermaceti processing, and selling of the final product were considered. In the late 18th–early 19th century, whaling expeditions could last up to five years (Starbuck 1878; Tower 1907; Irwin 2012). After a sperm whale was killed and once aboard the ship, the spermaceti was extracted from the head, separated from the oil and placed in barrels to be transported to manufactories. To be useable, spermaceti was refined by boiling. Purified spermaceti was then placed in barrels, and after a few more months of storage, the spermaceti was once again heated, hardened, and returned to bags to remove any final remnants of oil. The remaining waxy spermaceti was once again heated before being shaped into candles or other manufactured products. As a result, several years, estimated between 2 and 6 years, passed between the killing of a sperm whale and the end product. Chevreul, who began his investigations on animal fats in 1811 or 1813 (B. Bodo, personal communication), reported the properties of spermaceti for the first time in 1815 (Chevreul 1815), then in 1817 (Chevreul 1817), and finally in his first famous book

published in 1823 (Chevreul 1823). Chevreul abandoned fat chemistry in 1823 or 1824 when he was appointed director of dyeing at the Gobelins, the royal tapestry factory in Paris. Based on his publications, we can estimate that Chevreul obtained spermaceti materials between approximately 1811 and 1820. Taking into account both the whaling and commercial practices of the time and Chevreul's scientific work, it can be assumed that the spermaceti samples of the MNHN come from sperm whales caught at the beginning of the 19th century, probably between 1805 and 1815, i.e., AD 1810 \pm 5.

Sample Preparation and ^{14}C Measurements

Between 2 and 3 mg of spermaceti, blanc de baleine or cétine specimens were used. Except for two replicas cleaned by the standard ABA treatment, CO_2 was directly obtained from all the samples by combustion at 850°C for 5 hr in a sealed quartz tube with an excess of CuO (400–500 mg) and a 1-cm Ag wire. CO_2 was collected on a semi-automated rig (Dumoulin et al. 2017). CO_2 was then reduced to graphite with hydrogen over iron catalyst (Vogel et al. 1984). Radiocarbon measurements were performed by accelerator mass spectrometry (AMS) using the LMC14/ARTEMIS facility, a 3MV NEC Pelletron (Cottreau 2007; Moreau et al. 2020). Oxalic Acid II was used for normalization, and international intercomparison samples (FIRI H and FIRI I) for validation (Scott 2003). ^{14}C ages were calculated using the Mook and van der Plicht (1999) recommendations.

Calculation of MRE Values

Marine reservoir ages (R) were calculated by subtracting the expected radiocarbon age (in ^{14}C years) of the Muséum spermaceti specimens from the measured ^{14}C values.

Based on the above information (see the previous sections), a global date for the death of the sperm whales that produced the spermaceti samples was estimated at AD 1810 (with an uncertainty of \pm 5 yr). To take into account spermaceti turnover estimated at 10 years and to reflect the material variability (different sperm whales from various unknown locations), a shift of 5 years and additional uncertainties were applied. As a result, a date of AD 1805 \pm 15 was finally set, corresponding to an expected radiocarbon age of 157 \pm 15 yr BP.

ΔR values were calculated using the DeltaR function in the Marine Radiocarbon Database (<http://calib.org/JS/JSdeltar20/>) from the 14CHRONO Centre (Reimer and Reimer 2001) in September 2021. As the software does not enable errors on the collection year to be taken into account, two ΔR values ($\Delta\text{R}_{\text{min}}$ and $\Delta\text{R}_{\text{max}}$) were calculated using the two extreme values of the estimated death date of the sperm whales—AD 1790 (1805–15) and AD 1820 (1805+15)—as the independent age determination. An alternative approach, based on Macario et al. (2015), used Bayesian modeling in OxCal 4.4, by considering all the samples in a single phase, with ΔR undetermined over a range from –200 to 200 and including the calendar date as a C_Date of 1805 \pm 15 yr (see Supplementary material)

RESULTS AND DISCUSSION

For all the samples (except MNHN-CH-SC-2020-2572), AMS ^{14}C dating results were from 550 to 710 \pm 30 BP (Table 1). Calculated R values were obtained between 393 and 553 (\pm 34) ^{14}C yr and $\Delta\text{R}_{\text{min/max}}$ between –168 and 34 (\pm 60) ^{14}C yr. Bayesian modeling provided ΔR from –136 to 25 ^{14}C yr (95.4%) (Supplementary material). Taking into account the uncertainties, the results obtained by the two approaches are in agreement.

Table 1 ^{14}C results for the spermaceti, blanc de baleine and cétine specimens of the Muséum National d'Histoire Naturelle (National Museum of Natural History, Paris, France): age BP, R calculated for an expected radiocarbon age of 157 ± 15 yr BP, ΔR_{\min} (for an estimated death date of AD 1790) and ΔR_{\max} (for an estimated death date of AD 1820).

SacA N°	MNHN reference	MNHN description and <i>translation</i>	C mass (mg)	Age BP \pm 30	R \pm 34 ^{14}C yr	ΔR_{\min} (2σ) \pm 60 ^{14}C yr	ΔR_{\max} (2σ) \pm 60 ^{14}C yr
62418	CH-SC-2020-2564	Cétine cristallisée dans l'alcool <i>Crystalized cetine in alcohol</i>	1.46	675	518	-43	-2
62423	CH-SC-2020-2570	Cétine <i>Cetine</i>	1.30	580	423	-138	-97
62420	CH-SC-2020-2567	Spermaceti	1.55	655	498	-63	-22
62425	CH-SC-2020-2573	Spermaceti	1.60	680	523	-38	4
62421	CH-SC-2020-2568	Blanc de baleine dans l'alcool <i>Whale white in alcohol</i>	1.43	625	468	-93	-52
62419	CH-SC-2020-2565	Blanc de baleine purifié <i>Purified whale white</i>	1.62	710	553	-8	34
62422	CH-SC-2020-2569	Blanc de baleine <i>Whale white</i>	1.37	555	398	-163	-122
64310*			1.47	550	393	-168	-127
62424	CH-SC-2020-2572	Blanc de baleine <i>Whale white</i>	1.52	1125	968	408	449
64311*			1.31	1180	1023	463	504
		Mean value and standard deviation (all samples)			547 \pm 190	-14 \pm 190	27 \pm 190
		Mean value and standard deviation (all samples, except MNHN-CH-SC-2020-2572)			483 \pm 57	-78 \pm 57	-37 \pm 57

*These samples were pretreated by the ABA procedure.

For one sample (MNHN-CH-SC-2020-2572), we observed an older age at 1125 ± 30 BP. This sample was measured a second time, after ABA pretreatment, providing a similar result (1180 ± 30 BP). Two R values were obtained: 968 and $1023 (\pm 34)$ ^{14}C yr and ΔR values were from 408 to 504 (± 60) ^{14}C yr.

On average, ^{14}C dates on spermaceti samples (except MNHN-CH-SC-2020-2572) showed a mean offset R of 483 ± 57 yr (Table 1) from their estimated dates. These results are quite surprising as they reflect rather the global-average marine reservoir age of surface waters while sperm whales are deep divers. On the contrary, the result obtained for the sample MNHN-CH-SC-2020-2572 seems to be more consistent with deep-water values and may indicate a different origin or provenance for this specimen.

To the best of our knowledge, no MRE value has been reported for sperm whale spermaceti. The only comparison that can be made is with two values obtained for bones and published by Mangerud et al. in 2006. Recalculated from Marine20 (calib.org/marine/), R values for bones of sperm whales collected in Bretagne, France in 1890 and in North-Norway in 1896 are 278 and 328 ^{14}C years, respectively (Table 2). For other species, Mangerud et al. (2006) determined an average marine reservoir age (MRA) of 362 ± 38 yr relative to IntCal20/Marine 20 for various whales caught in Norway in the 19th century and Olsson (1980) a MRA of 315 ± 72 yr relative to IntCal20/Marine 20 for whales living near Sweden (Table 2). In total, 26 bones from different species of whales are recorded in the Marine20 database and provide a mean R_{whale} value of 350 ± 60 ^{14}C yr (Table 2). Other publications, not recorded in Marine 20, recommended using a ca. 200 yr marine reservoir correction for bowhead whales (*Balaena mysticetus*) from the Canadian Arctic (Dyke et al. 1996) or ca. 350 years for a 17th century Finback whale (*Balaenoptera physalus*) bone collected in Spitsbergen (Birkenmajer and Olsson 1998). Furze et al. (2014) provided reservoir offset values for bowhead whales corresponding to a MRA of 570 ± 95 ^{14}C yr, based on an exhaustive compilation of published marine mammal radiocarbon dates, both live-harvested materials and subfossils, from the Canadian Arctic Archipelago.

The measured deviations from the marine calibration curve (ΔR) for the spermaceti samples are from -168 to $34 (\pm 60)$ ^{14}C yr (Table 1) or from -136 to 25 ^{14}C yr (95.4%) depending on the calculation procedure used. These results differ from the values reported for two sperm whale bones, -241 ± 28 and -186 ± 23 ^{14}C yr, respectively and from most of the ΔR values obtained on bones from other species of whales recorded in the Marine20 database (Table 2). The mean ΔR values for the spermaceti samples (from -78 to 27 ^{14}C yr) are higher than the mean ΔR values for whale bones (-167 ± 52 ^{14}C yr).

Many factors can be put forward to explain these discrepancies: difference between spermaceti and bone turnovers, the unknown location and variability of sperm whales in the oceans, the industrial refining process used for spermaceti, and the impact of Chevreul's research work. Very little is known about the formation of spermaceti, which is a liquid composed of esters and acids, but it can be assumed that carbon integration differs from that which occurs in bones. In addition, unlike sperm whale bones, spermaceti is not a crude material, but has undergone many physical transformations, including several boiling/solidification cycles. And, lastly, some of the materials preserved at the Muséum are the result of Chevreul's experiments. For example, it is highly probable that the samples labeled "Cétine" or "Blanc de baleine purifié" were purified by Chevreul. Although we did not observe any significant difference in the ^{14}C results between purified and non purified

Table 2 Reservoir age and ΔR for bones of various species of whale extracted from the Marine20 database (Reimer and Reimer 2001). Location of collection is indicated by the longitude and latitude coordinates. Values for sperm whale (*Physeter catodon*) are indicated in bold.

Map no.	Genus	Species	Longitude	Latitude	Collection year	Reservoir age (yr)	ΔR (yr)	ΔR error (yr)	Reference
1075	<i>Physeter</i>	<i>catodon</i>	-5	48	1890	328	-186	23	Mangerud et al. (2006)
1074	<i>Physeter</i>	<i>catodon</i>	29.83	70.63	1896	278	-241	28	
1058	<i>Balaenoptera</i>	<i>physalus</i>	4.88	59.32	1865	405	-110	22	
1054	<i>Balaenoptera</i>	<i>acutorostrata</i>	5.08	60.27	1890	326	-188	34	
1055	<i>Balaenoptera</i>	<i>acutorostrata</i>	5.08	60.27	1869	297	-221	31	
1056	<i>Balaenoptera</i>	<i>acutorostrata</i>	5.08	60.27	1860	316	-205	32	
1064	<i>Eubalaena</i>	<i>glacialis</i>	5.08	60.35	1874	373	-134	32	
1069	<i>Lagenorhynchus</i>	<i>albirostris</i>	4.93	60.6	1885	375	-138	53	
1068	<i>Hyperoodon</i>	<i>ampullatus</i>	5.23	60.35	1887	370	-141	24	
1072	<i>Orcinus</i>	<i>orca</i>	5.23	60.35	1860	353	-168	29	
1073	<i>Orcinus</i>	<i>orca</i>	5.23	60.35	1887	361	-150	22	
1066	<i>Globicephala</i>	<i>melas</i>	4.95	60.82	1884	390	-124	21	
1065	<i>Eubalaena</i>	<i>glacialis</i>	5.23	60.58	1893	365	-155	23	
1057	<i>Balaenoptera</i>	<i>physalus</i>	5.02	61.58	1867	398	-119	24	
1071	<i>Megaptera</i>	<i>novaeangliae</i>	5.33	61.9	1901	340	-177	28	
1067	<i>Globicephala</i>	<i>melas</i>	10.67	59.88	1874	413	-94	24	
1059	<i>Balaenoptera</i>	<i>borealis</i>	22	70.63	1879	358	-155	20	
1060	<i>Balaenoptera</i>	<i>borealis</i>	22	70.63	1894	428	-93	50	
1061	<i>Balaenoptera</i>	<i>musculus</i>	27	71	1879	349	-164	23	
1062	<i>Balaenoptera</i>	<i>musculus</i>	27	71	1879	368	-145	21	
1070	<i>Lagenorhynchus</i>	<i>acutus</i>	31.08	70.38	1883	375	-141	32	
Mean (whales caught in Norway; Mangerud et al. 2006)						362 ± 38			
668	<i>Sibbaldus</i>	<i>muscul</i>	9.72	55.25	1939	332	-105	51	Olsson (1980)
673	<i>Balaenoptera</i>	<i>physalus</i>	12.4	56.98	1875	285	-222	45	
674	<i>Orcinus</i>	<i>orca</i>	11.43	58.28	1868	236	-281	100	
675		<i>sp.</i>	18.5	63.25	1657	406	-191	40	
Mean (whales caught in Sweden; Olsson 1980)						315 ± 72			
615	<i>Balaena</i>	<i>glacialis</i>	53.85	81.23	1936	160	-283	40	Forman (1997)
Mean and standard deviation R_{whale} and ΔR_{whale} (all data)						349 ± 59	-167 ± 52		

samples, it may be more accurate to select the “Spermaceti” samples for MRE values of this wax substance, that is to say, marine reservoir ages of 498 and 523 ± 34 ^{14}C yr and ΔR from -63 to 4 ± 60 ^{14}C yr. No other MRE values are reported in the literature for sperm whale spermaceti and further measurements on crude material would be necessary to confirm the results obtained here. Furthermore, it should be pointed out that in the case of these marine animals which travel all around the oceans during their long life, ΔR values cannot be related to a specific location but rather refer to spatially and temporally averaged values for that species.

CONCLUSION

Our study investigated the marine reservoir effect of spermaceti, a wax obtained from the head of the sperm whale. $R(t)$ and ΔR values were determined for eight samples collected by the French chemist Chevreur at the beginning of the 19th century and kept in the collection of the National Museum of Natural History, Paris, France. The $R(t)$ and ΔR values obtained in this study are higher than those reported in the literature for sperm whale bones collected in France and Norway at the end of the 19th century and also higher than almost all the values recorded for whales in the Marine20 database.

The values presented here are the first ever obtained for spermaceti. As they are based on museum specimens, there are some limitations such as the unknown location of the sperm whales caught for the spermaceti production as well as the possible chemical transformation of the material during Chevreur’s scientific work. These large uncertainties may limit the absolute dating of spermaceti wax objects and better-known reference materials would be necessary to improve accuracy.

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SUPPLEMENTARY MATERIAL

To view supplementary material for this article, please visit <https://doi.org/10.1017/RDC.2022.79>

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